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Document number: SD-WM-DP-052

Section 4 **of** 4

Title: PNL 325 Laboratory Single Shell Tank Waste Characterization Tank T-102 Cores 55 and 56 Data Package and Validation Summary

Date: 11-09-93 **Revision:** A000

Originator: KN Pool
Co: WHC

Recipient:
Co:

References: EDT-140716

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WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

PNL 325 LABORATORY SINGLE SHELL TANK WASTE CHARACTERIZATION, TANK T-102 CORES 55, 56 VALIDATION SUMMARY

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TABLE OF CONTENTS

T-105 CORES 55, 56

PACIFIC NORTHWEST LABORATORY

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The different formats are identified as follows:

		Number Series
1)	WHC	1, 2, 3 etc
2)	WHC	1A-1, 1A-2, etc (for 222-S & PNL Addendums to original Document)
3)	HASM	000006, 000007 etc
4)	PNL	B01-001, B02-002 etc

Lola R Webb 10/29/93

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Records Management Specialist

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LABORATORY CASE NARRATIVE

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INTRODUCTION

This Data Package contains the results obtained by the Pacific Northwest Laboratory (PNL), 325 Analytical Chemistry Laboratory (ACL) and the Chemical Processes Systems Section Staff for the characterization and analyses of Core 55 and the extrusion of Core 56 from Single-Shell Tank (SST) 241-T-102. The characterization and analysis requirements for Tank T-102 are outlined in the Tank Waste Remediation System-Tank Waste Characterization Plan (TWRS-TWCP, WHC-WM-SD-047 Rev. 1). Specific characterization activities are detailed in the Hanford Analytical Services Management (HASM) Statement of Work (WHC-SOW-93-0002 Rev. 0), the PNL Technical Project Plan (TPP, dated June 30, 1993), and the PNL, Quality Assurance Project Plan (QAPjP, ALO-003 Rev. 1). Due to the low sample recovery from Cores 55 and 56 a Letter of Instruction (LOI) (9355091, dated July 8, 1993) was issued by HASM and Westinghouse Hanford Company (WHC), Analytical Evaluation and Reporting (AE&R) to specifically define the characterization requirements for the recovered material. Test Instructions prepared by the PNL Project Manager, define the specific preparation and analyses.

Three casks were shipped from WHC Tank Farms Operations to the PNL High-Level Radiation Facility (HLRF) on April 1, 1993. The cask seals were intact and the information on the seals matched the chains-of-custody. The cask (C1033) containing the Field Blank was opened on April 16, 1993. The liner and liner plug were contaminated with beta-gamma activity, which was unexpected since the cask contained a water Field Blank. The liner was decontaminated and the Field Blank sample was drained from the sampler. No contamination was detected on the sampler.

The second cask (1001C) was opened on April 20, 1993 and found to be empty. The chain-of-custody showed that a sampler containing Core 55 should have been in this cask. WHC was notified and a PNL Nonconformance Report (NCR No. 93-022) was issued based on non-receipt of the sample. A second shipment (cask number 1004C) containing Core 55 was received on May 4 and extruded on May 14, 1993. Approximately five inches of solid material, 80.59 grams, and less than 10 ml of liquid were obtained from this sampler. The recovery for Core 55 was 65% of the expected volume.

The cask (C1032) containing Core 56 was turned into a horizontal position and seated against the door of the hot cell. Contaminated water from the cask leaked into the hot-cell, the hot-cell scoop, and down the cell face. The amount of water that leaked from the cask could not be accurately determined, but was estimated to be over one liter. Core 56 was extruded from the sampler on April 21, 1993. Core 56 contained 8.4 grams of solid material; the recovery for Core 56 was less than 10% of the expected volume.

Photographs of the extruded material can be found in Figures 1-1 through 1-3. After extrusion, the Core 55 and 56 material was placed in glass jars, sealed and stored in the HLRF. Characterization activities on the T-102 samples were delayed until the PNL TPP and QAPjP were completed, and the LOI was issued by HASM and WHC AE&R. Core 55 was sub-sampled for rheologic analysis. The remainder of the Core 55 material was homogenized by hand and treated as a single core composite sample. The ICP data obtained from the Core 55 homogenization test samples indicated that additional homogenization was necessary. The sample was transferred to the Shielded Analytical Laboratory (SAL) for the additional homogenization. Fine dark particles were observed in the sample during the homogenization in the SAL. A mortar and pestle was used to grind the Core 55 material and produce a more homogeneous sample. The core composite was not re-sampled for homogenization test analyses due to the limited amount of sample available. The core composite was sub-sampled and aliquots analyzed for the core composite suite of analyses, in the order of priority listed in the LOI. All of the material from Core 56 was transferred to the SAL for archive; no analyses were performed on this core.

A Hot Cell Blank was generated before extrusion of Core 55. This sample and the T-102 Field Blank were analyzed for the suite of analyses required by the TWRS-TWCP.

The ACL sample numbers assigned to the samples and sub-samples are listed in Table I-1. This table shows the ACL sample numbers and analyte by sample preparation method.

The data within this package are divided into three groups: Physical Testing, Inorganic Analysis, and Radiochemical Analysis. All chemical analysis data are reported on a wet-weight basis. That is, no corrections have been made for the water content in the sample. Sample preparations were performed in duplicate. The quality control (QC) requirements for each sample are defined in the Test Instructions. Samples were prepared and analyzed as a batch where feasible. A minimum number of QC samples were analyzed in each batch and all QC data are included in this data package. It should be noted that all of the QC requirements in the QAPjP could not be met for the inorganic preparation and analyses. A PNL Deficiency Report (DR-93-033) was issued and is included in Appendix A2 of the data package.

Table I-1: T-102 Core 55, PNL-ACL Sample Numbers

RHEOLOGY	CORE 55 COMPOSITE 93-08755	Rheology Sample
ACID DIGESTION	93-08755-A1	Acid Digest. Sample (ICP)
	93-08755-A2	Acid Digest. Duplicate
	93-08755-A3	Methods Blank (minimum 1 per batch)
	93-08755-A4	Matrix Spike (ICP)
	93-08755-A5	Spike Control (ICP)
WATER LEACH	93-08755-C1	Water Leach Sample *
	93-08755-C2	Water Leach Duplicate
	93-08755-C3	Methods Blank (minimum 1 per batch)
	93-08755-C4	Matrix Spike (ICP and IC)
	93-08755-C5	Spike Control
	93-08755-C6	Post Digestion Spike (Radionuclides)
MERCURY	93-08755-D1	Mercury Sample
	93-08755-D2	Mercury Duplicate
	93-08755-D3	Methods Blank (minimum 1 per batch)
	93-08755-D4	Matrix Spike
	93-08755-D5	Spike Control
TOTAL CYANIDE	93-08755-G1	Total CN Sample
	93-08755-G2	Total CN Duplicate
	93-08755-G3	Methods Blank (minimum 1 per batch)
	93-08755-G4	Matrix Spike
	93-08755-G5	Spike Control
HOMOGENIZATION TEST FUSION DISSOLUTION	93-08755-H1T	Homogenization Test Fusion Sample - Top
	93-08755-H2T	Homogenization Test Fusion Duplicate - Top
	93-08755-H3T	Homogenization Test Fusion Blank
	93-08755-H1B	Homogenization Test Fusion Sample - Bottom
	93-08755-H2B	Homogenization Test Fusion Duplicate - Bottom
FUSION DISSOLUTION	93-08755-H1	Fusion Sample (ICP & Radchem **)
	93-08755-H2	Fusion Duplicate
	93-08755-H3	Methods Blank (minimum 1 per batch)
	93-08755-H6	Post Digestion Spike (ICP and Radchem)
CARBON	93-08755-J1	Carbon Analysis Sample (TC/TIC/TOC).
	93-08755-J2	Carbon Analysis Duplicate
	93-08755-J4	Matrix Spike
WT% SOLIDS	93-08755-K1	Wt. % Solids Sample
	93-08755-K2	Wt. % Solids Duplicate
DIRECT	93-08755-M1	Wt. % Oxides, Density, pH, OH- and DSC/TGA Sample
	93-08755-M2	Wt. % Oxides, Density, pH, OH- and DSC/TGA Duplicate
	93-08755-M3	Methods Blank
PARTICLE SIZE	93-10374-M1	Particle Size Sample
	93-10374-M2	Particle Size Duplicate
FUSION OF WATER LEACH RESIDUAL SOLIDS	93-08755-CH-1	Caustic Fusion of Water Leach Residual Solids Sample (GEA)
	93-08755-CH-2	Caustic Fusion of Water Leach Residual Solids Duplicate (GEA)
	93-08755-CH-3	Methods Blank (minimum 1 per batch)
	93-08755-CH-6	Post Digestion Spike (GEA)

* Water Leach: IC, ICP, NH3, TOC/TIC/TC, GEA, Total Alpha, Total Beta, C-14, H3, Cr(VI), TDS.

** Fusion Radchem: Total Alpha, Total Beta, GEA, U, Tc, Sr/Y, Alpha Pu, Np, Am/Cm, U Isotopics.

Table I-1: T-102 Core 55, PNL-ACL Sample Numbers Cont'd

	T-102 Field Blank	HLRF Hot-Cell Blank	HLRF DIW	
Direct	93-05874-P-1	93-09774-P-1	93-09804-P-1	Sample for TOC, CN-, pH, OH-, DSC/TGA
	93-05874-P-2	93-09774-P-2	93-09804-P-2	Duplicate
	93-05874-P-4	93-09774-P-4		Matrix Spike (TOC and CN-) 1 per batch
	93-05874-P-5	93-09774-P-5		Spike Control (CN- only) 1 per batch
Direct Filtered	93-05874-Q-1	93-09774-Q-1	93-09804-Q-1	Sample for IC
	93-05874-Q-2	93-09774-Q-2	93-09804-Q-2	Duplicate
		93-09774-Q-4		Matrix Spike (1 per batch)
				Spike Control (1 per batch)
Direct Acidified	93-05874-R-1	93-09774-R-1	93-09804-R-1	Sample for ICP, NH3, GEA, Total Alpha & Beta
	93-05874-R-2	93-09774-R-2	93-09804-R-2	Duplicate
	93-05874-R-4	93-09774-R-4		Matrix Spike for ICP (1 per batch)
	93-05874-R-5	93-09774-R-5		Post-filtered matrix spike for Rad Chem (1 per batch)

SECTION 1

PHYSICAL DATA

Figure 1-1: T-102 Core 55, Photograph During Extrusion

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Figure 1-2: T-102 Core 55, Photograph

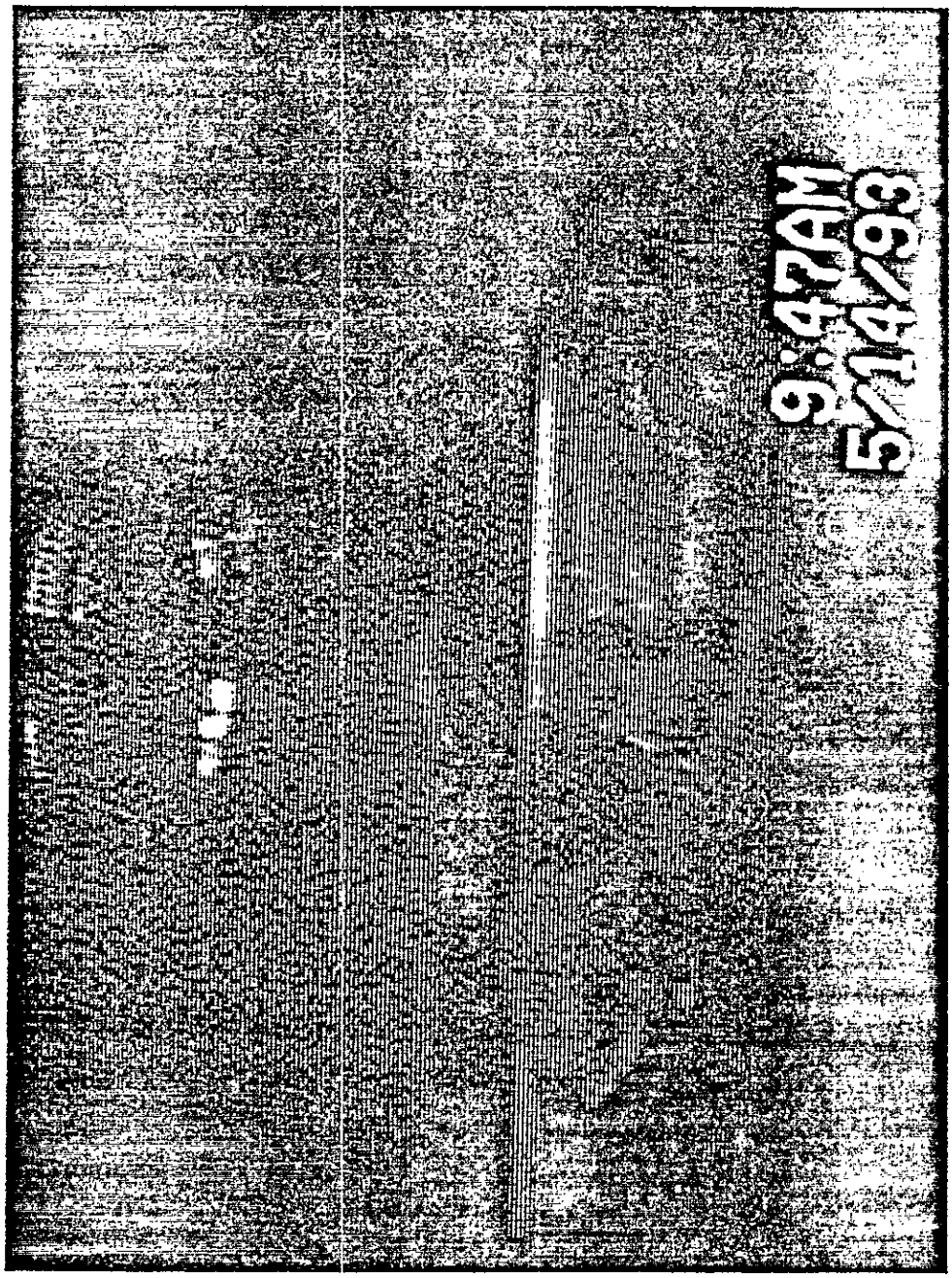

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Figure 1-3: T-102 Core 56, Photograph

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EXTRUSION AND SAMPLE PREPARATION

Description of Extruded Segments: On April 1, 1993, the HLRF staff received shipment S93-008 from WHC. This shipment contained three casks: 1001C, C1032 and C1033. The chains-of-custody stated that these casks contained two core samples from Tank 241-T-102 and a field blank (water sample). The chains-of-custody for these samples were signed by TK Andrews, HLRF Supervisor. The seals on each of the casks were intact, and the data on the seals matched the chains-of-custody.

On April 16, 1993, cask C1033 (sample 93-008) was opened. This cask contained the field blank; therefore, this cask and its contents should not have been contaminated. The liner and liner plug were found to be contaminated with 40,000 dpm and 25,000 dpm of beta-gamma activity, respectively. Fortunately, no contamination was detected on the sampler. The liner was decontaminated and the field blank was drained from the sampler.

On April 20, 1993, casks 1001C and C1032 were opened. Cask 1001C was empty. The chain-of-custody associated with this cask stated that sampler 91-148 containing sample 93-009 was loaded into this cask. No sampler or liner was present. A PNL Nonconformance Report (PNL-022) was issued based on non-receipt of sample.

Upon placing cask C1032 in a horizontal position and seating it against the door of the hot-cell, water leaked into the scoop, the hot-cell and down the cell face. The amount of water which was in the cask could not be accurately determined, but it was estimated that one liter of water leaked into the hot-cell, 150 ml of water leaked into the scoop and >50 ml of water leaked down the cell face. The radioactive contamination from the water which leaked down the cell face was approximately 4000 dpm. The cell port read approximately 40,000 dpm after the liner and sampler were transferred into the hot-cell.

Core 56 the sample from cask C1032 (sample 93-010) was extruded from the sampler on April 21, 1993. The sampler was found to contain 4 ml of material based on an approximate sample length of 1.25 inches and diameter of 0.5 inches. The chain-of-custody estimated the sample volume at 70 ml. Using this estimated

volume, the recovery for Core 56 was less than 10%. This sample contained 8.4 grams of solids and no drainable liquids. The limited sample available from this core precluded analysis of physical and rheological properties.

On May 4, 1993, the HLRF staff received sample 93-009 (Core 55) in cask 1004C. This was shipment S93-009. Sample 93-009 was reported as being shipped to PNL in cask 1001C on April 1, 1993, but this cask was empty upon arrival; therefore, strict chain-of-custody was lost on this sample.

Core 55 was extruded on May 14, 1993, and 45 ml of sample was obtained from this sampler. The estimated length for this core sample as reported on the chain-of-custody was seven inches (69 ml); therefore, a 65% recovery was obtained for this core. The mass of this sample was 80.59 grams. Minimal drainable liquid (less than 10 ml) was associated with this sample; therefore the drainable liquid was not separated from the solids.

The 45 ml sample volume is based upon the mass of the sample on the extruder tray and the density of the core sample measured after the extrusion process. The initial recovery reported immediately after the extrusion process (70%) was based on an estimated sample volume of 49 ml. This volume was calculated from the measured length of the sample (5 inches) and an assumed sample diameter of 0.875 inches. This sample radius was based on the diameter of the sampler.

The WHC sample numbers, mass, volume, and density for the two cores and the field blank are given in Table 1-1.

Table 1-1: T-102 Core 55, Unhomogenized Core Data

SAMPLE ID	WHC SAMPLE No.	MASS (g)	VOLUME (ml)	DENSITY (g/ml)
Field Blank	93-008		187	
Core 55	93-009	80.59	45	1.79
Core 56	93-010	8.42	4	2

Of the five inches of solids obtained from Core 55, the top two inches of the sample were dry and crumbly. This two inch portion of the sample was brown with streaks of white. The next two inches were a white sticky sludge with brown streaks. The bottom inch of the segment had a similar consistency to the top two inches (dry and crumbly), but the entire sample was brown. The 8 grams of Core 56 sample were brown with a dry granular texture.

The 8 grams of Core 56 was archived without any homogenization being performed. The extruded sample from Core 55 was split lengthwise along the extrusion tray. One side of the sample was used for rheological analysis. The other side (Core 55 Composite) was used for the remaining analyses.

Two sub-samples for particle size and thermal analysis were taken from the Core 55 Composite prior to homogenization. The remaining 26 grams of the Core 55 Composite was homogenized using hand mixing techniques. Mechanical mixing was not used, because too much of the sample would be lost in the mixing procedures. Homogenization check samples were taken from the top and bottom portions of the homogenized material.

The homogenization check samples were prepared for ICP by Ni/KOH fusion. The melt was redissolved in deionized water and HCl. This sample was analyzed by ICP to determine if the sample was sufficiently homogenized. The relative percent differences (RPD's) between the sample and duplicate from each sample and between the top and bottom samples were outside the acceptable range. The RPD is calculated as the variance between two analyses divided by the average of the two analyses. The large variances indicated that heterogeneity existed within each sample.

The Core 55 Composite was further homogenized in the SAL by crushing and grinding the sample with a Diamonite mortar and pestle. Prior to crushing small dark specks were observed throughout the otherwise whitish solids. These dark specks were magnetic.

The solids which were crushed were very dry in appearance, but electrostatic clinging of particulates to container surfaces was very minimal.

After crushing the sample, the dark specks were no longer observed; but after water leaching a portion of the crushed solids, the dark specks were again visible. It appears that crushing coated the specks with fine white particles.

A Hot-Cell Blank was prepared between the extrusions of Core 56 and Core 55. This blank was prepared by washing the cleaned extrusion tray with approximately 250 ml of distilled water. The distilled water was obtained from the same source as all water used in the hot-cell operations. The wash solution was captured in a 250 ml borosilicate jar and transferred to the SAL.

Sample Preparation: A single, blended Core 55 composite solids sample, a Hot Cell Blank and a Field Blank together comprise the work scope for Tank T-102. They were transferred from the HLRF to the SAL. Due to the high level of radioactivity associated with the solids from Tank T-102, all of the analytical preparations were completed in the hot cell. The Field and Hot Cell Blanks were radiologically low-level but were processed in the hot cell to allow comparability with the sample.

Table 1-2 lists the procedures that were used to prepare Tank T-102 samples for the suite of requested analyses. Also included, are the procedures that were used to conduct a limited number of in-cell analytical determinations.

Table 1-2: T-102 Core 55, SAL Procedure List

PNL Procedure Number	Procedure Title
PNL-ALO-101, Rev. 1	Acid Digestion for Metals Analysis
PNL-ALO-102, Rev. 0	Fusion of Hanford Tank Waste Solids
PNL-ALO-103, Rev. 1	Water Leach of Sludges, Soils, and Other Solid Samples
PNL-ALO-213, Rev. 0	Mercury in Water, Solids, and Sludges by Manual Cold Vapor Technique
PNL-ALO-285, Rev. 0	Total Cyanide by Remote Microdistillation and Argentometric Titration
PNL-ALO-320, Rev. 0	Method for Extractable Organic Halides (EOX) in Solids
PNL-ALO-381, Rev. 0	Determination of TC,TOC, and TIC in Radioactive Liquids, Soils, and Sludges by Hot Persulfate Method
PNL-ALO-482, Rev. 0	Determination of Carbon-14 in Radioactive Liquids, Solids, and Sludges
PNL-ALO-501, Rev. 0	Laboratory Procedure for Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges
PNL-ALO-504, Rev. 1	Percent Solids Determination of Hanford Tank Waste Sludges
SW-846 Method 9045-B	Soil and Waste pH

Sample preparations/distributions of the blended Core 55 solids involved:

1) Water leaching for IC, ICP, NH_3, TOC/TIC/TC, Cr(VI), TDS, pH, OH, GEA, Total Alpha, Total Beta, C-14, H-3.

2) Ni/KOH fusions for ICP, GEA, Total Alpha, Total Beta, AEA for Pu,Np,Am, Uranium, Tc-99, Sr/Y-90, and Pu/U isotopics.

3) Acid digestions or distillations for ICP, CN and Hg.

4) The distribution of direct sub-samples for DSC/TGA and Weight Percent Oxides.

Following the water leaching process, the undissolved sample residue was dried and weighed. Portions of the dried solids were fused (Ni/KOH) for GEA.

Predigestion spikes were performed for ICP metals, IC anions, cyanide, carbon, C-14 and mercury analyses only. Post digestion spiking was done at the laboratory bench by the functional group performing the analysis.

Bulk Density, Weight Percent and Total Dissolved Solids determinations were completed in-cell. A 1:5 (not 1:1) water contact was made for pH and OH^- due to limited sample availability.

The Tank T-102 work scope included both a Field and Hot Cell Blank. Per Test Instruction, portions of each blank were acidified with nitric acid and distributed to the laboratories for ICP, NH_3, GEA, Total Alpha and Total Beta. A portion of each blank was filtered (0.45 micron) for ion chromatography. Direct blank portions were distributed for TOC, CN, pH, OH and DSC/TGA analyses.

During sample preparation, the SAL made deliberate minor deviations to sample preparatory procedures for one or more of the following reasons:

1) Insufficient sample was available to conduct the analyses per procedure while maintaining the level of quality control requested.

2) Sample weights and/or final volumes were reduced to facilitate waste minimization.

3) Sample weights and/or final volumes were altered to increase the concentration of certain analytes of interest. This was done to meet the procedural concentration ranges needed to perform the analyses.

Sample sizes and final volumes for all sample preparations are documented on the Sample Preparation Sheets included in Appendix B5. Table 1-3 lists the sample preparatory procedure deviations performed during the processing of Tank T-102.

Table 1-3: T-102 Core 55, Sample Preparation Procedural Deviations

ALO Number	Prep Method	Sample Size Deviation	Sample Volume Deviation	Reagent Deviation	Observed Effect
93-08755-A	Acid	Yes	No	No	None
93-08755-C	Water	Yes	Yes	No	None
93-08755-D	Acid (Hg)	No	Yes	No	None
93-08755-G	Acid (CN)	Yes	Yes	No	None
93-08755-K	Wt% Solids	Yes	N/A	N/A	None
93-08755-M	Water	Yes	Yes	N/A	None

THERMAL ANALYSIS

Differential Scanning Calorimetry (DSC) and Scanning Thermogravimetry (STG) were performed in duplicate on the unhomogenized material from Core 55. DSC and STG were also performed on the field blank, hot cell blank, and the water used to prepare the hot cell blank. These two thermal analysis techniques are useful in determining the thermal stability and reactivity of a material. DSC measures heat released or absorbed while the temperature of the sample is increased at a constant rate. Data generated by the DSC analysis is often used to measure thermal decomposition temperatures, heats of reaction, reaction temperatures, melting points and solid-solid transition temperatures. STG measures the mass of a sample while the temperature of the sample is increased at a constant rate. The STG data is used to measure thermal decomposition temperatures, water

contents and reaction temperatures. Both methods can be modified to measure isothermal changes in the material and provide complimentary information.

The calibration of the DSC and STG instruments were checked before running these samples. An indium standard was run on the DSC to check the temperature and enthalpy calibrations. The balance calibration of the STG was checked with a 100 mg standard weight, and the temperature calibration was checked with alumel and perkalloy curie point magnetic transition standards. The temperature and enthalpy calibration checks were all within 2°C and 0.1 calories per gram of their reported values, and the balance calibration was within 0.01 mg.

The results from the DSC and STG analyses of the Core 55 sample are reported in Table 1-4 and 1-5, respectively. The temperature range of the DSC scan was from ambient to 500°C, with a scan rate of 5°C per minute. Two endothermic transitions were observed in this temperature range. A minor endothermic region was observed between 70°C and 100°C. The onset temperature of this event was 76°C, with an enthalpy of 2.4 calories per gram of sample (10 J/g). An associated mass loss of between 1% and 2% was observed in the STG. This mass loss compares well with the 99.1 weight percent solids measured on this same sample in the SAL (see Table 1-7). The temperature of this event and the mass loss observed by the STG analysis suggests the loss of free water.

The major endothermic region was observed between 200°C and 365°C. This endothermic event includes at least two unresolved peaks. The onset temperature of this event was 255°C. This onset temperature is based on the major peak which is actually the second peak in this region; therefore, the first reaction in this region begins at a lower temperature than the onset temperature. The onset temperature for this reaction is estimated to be about 219°C. The enthalpy of transition region averaged 315 calories per gram of sample (1320 J/g). The STG analysis showed this transition was accompanied by a 24% loss in mass. This endotherm and its associated mass loss is probably due to the decomposition of aluminum hydroxide to produce aluminum oxide and water. Based on the mass loss observed and the aluminum concentration measured on the fused sample, 1.2 moles of water were lost during this transition per mole of Al.

A third endothermic event was noted on the DSC and STG analyses starting at approximately 410°C and running beyond the end of the analysis (500°C for DSC and 550°C for STG). The STG observed a 7.3% mass loss before the upper temperature limit of the instrument halted the analysis. No analysis can be performed on the front end of the peak observed by DSC.

Analysis of the field blank by DSC and STG was conducted as per the letter of instruction. The DSC showed a large endothermic event from 40°C to 110°C. Since these samples are 100% H_2O, the energy associated with this transition exceeded the sensitivity range of the system and a flat top was observed on peak of this transition. This event, the vaporization of water, was observed on the STG as a mass loss of 100%. No other events were detected by either instrument.

Table 1-4: T-102 Core 55, Differential Scanning Calorimetric (DSC) Data

	Transition #1			Transition #2		
Run	Enthalpy (cal/g)	Onset (°C)	Range (°C)	Enthalpy (cal/g)	Onset (°C)	Range (°C)
1	2.6	76	70-103	315	255	200-365
2	2.3	76	70-111	323	255	190-380

Table 1-5: T-102 Core 55, Scanning Thermogravimetric (STG) Data

	Transition #1		Transition #2		Transition #3	
Run	Range (°C)	Mass Loss (&)	Range (°C)	Mass Loss (%)	Range (°C)	Mass Loss (%)
1	30-190	2.0	190-370	23.5	370-545	7.4
	30-190	1.0	190-370	25.2	370-545	7.2

PHYSICAL PROPERTIES, SETTLING BEHAVIOR AND RHEOLOGICAL PROPERTIES

Tests performed on Core 55 include weight percent solids, weight percent oxides, particle size, sample density, centrifuged supernate density and solids density, settling behavior, weight percent centrifuged solids and volume percent centrifuged solids. Shear stress as a function of shear rate (viscosity) could not be performed on the as-received core sample. Viscosity measurements are applicable to materials which flow; not to dry non-fluid samples. The limited sample available from Core 55 also precluded analysis of penetration resistance and shear strength. The current penetrometer requires a sample at least 0.875 inches in diameter and 1.5 inches deep to obtain a valid measure of penetration resistance. The current shear vanes available for shear strength determinations require a sample 0.5 inches deep and 0.857 inches in diameter.

Shear stress as a function of shear rate, settling velocity and volume percent settled solids were performed on 1:1 and 1:3 sample to water dilutions. Shear stress as a function of shear rate was performed at both 25°C and 90°C.

Experimental Procedures

Density: The samples were placed in preweighed, volume-graduated, centrifuge tubes where they were weighed and then centrifuged for one hour at greater than 1000 gravities to remove voids. This ensured accurate volume measurements and allowed division of the sample mass by sample volume to obtain density. The density of the centrifuged supernate was obtained by transferring the centrifuged supernate to a preweighed graduated cylinder. The volume and mass of the sample were recorded and the density was calculated by dividing the mass of the sample by the volume of the sample.

Solids Settling Rate and Volume Percent Settled Solids: Settling rates and volume percent settled solids measurements were conducted in preweighed, volume-graduated, centrifuge tubes. The cross-sectional area in the upper portion of the centrifuge tubes was constant thus allowing the conversion of settling rate data from ml/hr to cm/hr. After settling rates were determined, the volume

percent settled solids were calculated by dividing the final settled solids volume by the total sample volume.

Weight Percent Solids and Weight Percent Oxides: Samples were placed into preweighed vials, weighed and allowed to air-dry overnight to remove free liquid to prevent splattering in the oven. The samples were then transferred to a muffle furnace or drying oven at 105°C where they were dried for 24 hours. The dried samples were removed from the oven, placed in a desiccator to cool to room temperature, reweighed, and the weight percent total solids was calculated.

For determination of weight percent oxides, the samples were placed into preweighed crucibles, weighed and allowed to air-dry overnight to remove free liquid to prevent splattering in the oven. The samples were then transferred to a muffle furnace at between 1000°C and 1050°C for 30 minutes. The calcined samples were removed from the oven, placed in a desiccator to cool to room temperature reweighed, and the weight percent oxides was calculated.

Particle Size: Particle size distribution was measured in duplicate on unhomogenized material from Core 55. A Brinkmann Model 2010 Particle Size Analyzer was used to determine the distribution of particle sizes. The analysis was performed according to PNL technical procedure PNL-ALO-530 Revision 0, "Particle Size Distribution By Laser Scanning (Time of Transition)." The Brinkmann particle size analyzer determines particle size in the range of 0.5 μm to 150 μm by measuring the time required for a rapidly moving laser beam to traverse selected particles maintained in a stirred suspension. A glass sphere reference (Duke 147) with a nominal mean particle diameter of 20 μm is measured with each sample batch.

Shear Stress versus Shear Rate: Dilutions were analyzed in duplicate for shear stress as a function of shear rate using a Bohlin CS viscometer modified for glove box operation. Concentric cylinders with a 25 mm diameter inner cylinder and a 2.5 mm gap between the cylinders were the measuring geometry used with a C25 measuring sensor. Shear stress as a function of shear rate data was obtained by measuring the shear stress produced at a specific shear rate. Calibration checks were made with certified 50 cP and 10 cP viscosity standards to ensure

that the viscometer was operating properly. Technical Procedure PNL-ALO-501, "Laboratory Procedure for Measurement of Physical and Rheological Properties of Solution, Slurries and Sludges" was used to perform these measurements.

Results

Settling Behavior: The physical properties of the unhomogenized Core 55 sample are summarized in Table 1-6.

Table 1-6: T-102 Core 55, Physical Properties Summary

Physical Properties	As-Received	1:1 Dilution	1:3 Dilution
Settled Solids (vol%)	100	15.7	8.3
Centrifuged Solids			
Volume Percent	96		
Weight Percent	97		
Density (g/ml)			
Sample	1.79	1.11	1.05
Centrifuged Supernate	1.1		
Centrifuged Solids	1.8		
Total Solids (wt%) [a]	72.3		
Oxides (wt%) [b]	65.7		

(a) This weight percent total solids value is the measured value of the sample used for the rheological and settling properties of the waste. This is not the sample used for the chemical, radiochemical, energetics, or weight percent oxides. Additional data is given in Table 1-7.

(b) Weight percent oxides was measured on the homogenized sample from the SAL.

Settling was not observed for the undiluted Core 55 sample, but settling was observed on the 1:1 and 1:3 sample:water dilution. If this sample were

composed of 100% insoluble solids, the packing density of the settled solids was the same for the undiluted and diluted sample, and the added water was not associated with the solid particles; the expected vol% settled solids for the 1:1 dilution would be near 36%. The value of 15.7% for the 1:1 dilution indicates that at least half the solids in Core 55 are soluble. The water leach data indicates much lower solubility than was observed on the 1:1 dilution. The inorganic data indicates that the primary compound in the sample is aluminum hydroxide which is amphoteric (exhibits both basic and acidic properties). The increased solubility in the 1:1 dilution compared to the water leach (100:1 dilution) is due to the differences in the pH. At the higher pH present in the 1:1 dilution, the equilibrium is pushed toward the formation of $Al(OH)_4^-$ where in lower pH solutions like the water leach solution $Al(OH)_3$ is favored.

The data in Table 1-6 shows a two-fold decrease in the vol% settled solids between the 1:1 and the 1:3 dilution indicating that the solids remaining after the 1:1 dilution are essentially insoluble or that the pH has been decreased enough to decrease the solubility of the remaining solids.

The volume percent settled solids as a function of time for both the 1:1 (dilution 1) and 1:3 (dilution 2) dilutions are reported in Figure 1-4. Duplicate measurements for each of the dilutions are plotted in this figure. Significant settling for both dilutions were observed over 30 hours, but the settling velocities decreased sharply over the first hour as reported in Figure 1-5.

Weight Percent Solids: The weight percent solids of the Core 55 sample was measured in duplicate at three different times. The data from these three analyses plus the weight percent solids measured by STG are given in Table 1-7. Weight percent dissolved solids were not measured on the Core 55 sample, because limited supernate was available.

The weight percent solids was initially measured on two portions of the extruded Core 55 sample. The two sub-samples were taken from different portions of the segment which appeared to have different amounts of moisture; therefore, there is significant variability in the measured weight percent solids of these

two sub-samples. During the extrusion process, the core sample was split into two different samples, a rheology sample and a sample for chemical and radiochemical characterization. Both of these samples were stored in the hot cell for a period of four months before any further analyses were begun. The chemical and radiochemical characterization sample was then homogenized and transferred to the SAL where the weight percent solids of this sample was analyzed. A sub-sample of the SAL sample was used to measure the energetics of the waste using DSC and STG. The initial water loss data from the STG is also presented in Table 1-7. Significant drying of the SAL sample occurred during processing of the sample and/or during the time it was stored in the hot cell. This same drying process was not observed in the rheology sample.

Table 1-7: T-102 Core 55, Weight Percent Total Solids

	Wt% Solids			RPD (%)
	sample	duplicate	average	
Extrusion Samples	74.2	69.0	71.6	7.2
Rheology Sample	72.1	72.4	72.3	0.4
SAL Sample	99.11	99.12	99.12	0.01
STG Sample	98.0	99.0	98.5	1.0

Particle Size: Graphs of the probability number density and volume density for Core 55 sample and the Duke standard are given in Appendix C4. The median and mean particle sizes of the Core 55 sample based on volume density are 35 μm and 36 μm respectively, with 90% of the particles between 10 μm and 60 μm. The median and mean particle size of the Core 55 sample based on number density are 0.93 μm and 2.35 μm respectively, with 90% of the particles less than 4 μm and 99.99% less than 60 μm. Analysis of the duplicate confirmed these results.

Rheological Properties

Both the 1:1 and 1:3 dilutions exhibited some dilatant behavior over the measured range (0 s^{-1} to 500 s^{-1}). Dilatancy generally occurs in concentrated

suspensions which tend to gel upon mixing. The dilatant behavior is identified by an increase in viscosity with increasing shear rate. Because of the low viscosities observed for these two dilutions, the significance of this dilatant behavior is limited. None of the dilutions, 1:1 or 1:3, exhibited yield points. This rheology data was fit to the power law equation (see Equation below), and the curve fit parameters are given in Table 1-8.

$$\tau = K\gamma^n$$

where K = consistency parameter,
γ = shear rate
n = flow behavior index.

Table 1-8: T-102 Core 55, Power Law Fit Parameters for 1:1 and 1:3 Dilutions

Dilution	Temperature (°C)	Run	Consistency Parameter (Pa·s)	Flow Behavior Index
1:1	25	1	0.039	1.8
1:1	25	2	0.078	1.6
1:1	90	1	0.10	1.5
1:1	90	2	0.082	1.5
1:3	25	1	0.13	1.5
1:3	25	2	0.10	1.6
1:3	90	1	0.13	1.4
1:3	90	2	0.14	1.4

The viscosity of the 1:1 dilutions at ambient temperature varied between 1 cP and 4 cP over a shear rate range of 50 s^{-1} to 400 s^{-1}. At 90°C, the viscosity of the 1:1 dilutions varied between 0.5 cP and 2 cP over a shear rate range of 50 s^{-1} to 400 s^{-1}.

The viscosity of the 1:3 dilution at 25°C increased from 1 cP to 3.5 cP in the shear rate range from 50 s^{-1} to 400 s^{-1}. At 90°C, the viscosity of this dilution increased from 0.6 cP to 1.3 cP over the same shear rate range.

Plots of shear stress and viscosity as a function of shear rate for the dilutions are given in Appendix C8.

Figure 1-4: T-102 Core 55, Volume Percent Settled Solids for the 1:1 and 1:3 Sample to Water Dilutions

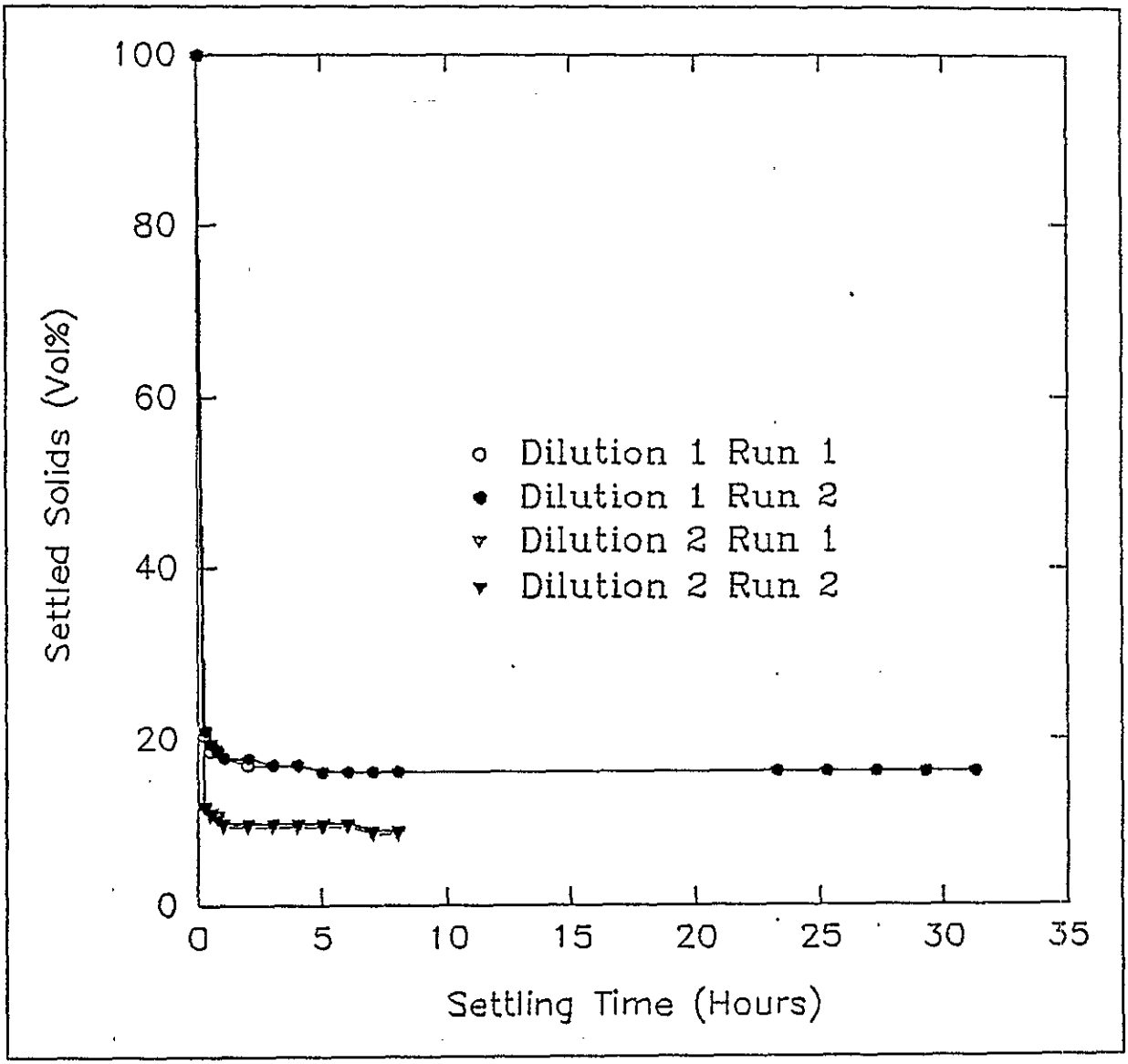

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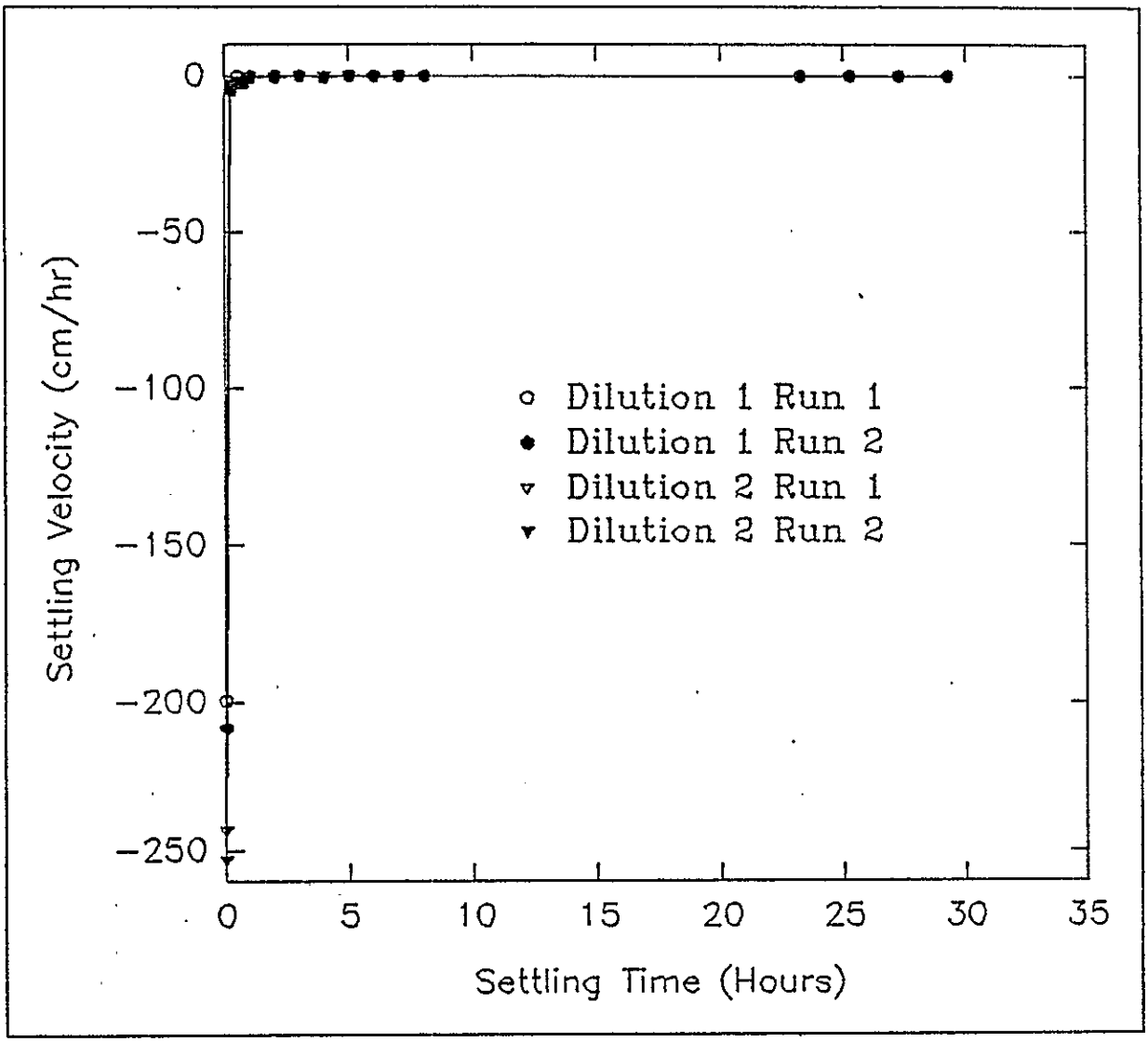
Figure 1-5: T-102 Core 55, Settling Velocity for the 1:1 and 1:3 Sample to Water Dilutions



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ADDENDUM 1 REV. 0

SECTION 2

INORGANIC CHEMISTRY

ICP Analysis -- Fusions Results

ICP analyses were performed on fusions prepared from Core 55 composite material. The samples were prepared following procedure PNL-ALO-102, "Fusion of Hanford Tank Waste Solids" (KOH fusions in Ni crucibles), and analyzed following procedure PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry." The fusions and subsequent melt acid dissolutions were performed in the SAL and the digestates transferred to the Inorganic Analysis Group for ICP analysis. All ICP analyses were performed on a Jarrell-Ash ICP system with interelement corrections for spectral interferences being performed on-line.

The fusion results for the composite sample, duplicate, and blank are reported along with the post spike QC results. The composite samples were analyzed at both a 2x and 10x dilution with the corresponding percent difference (%D) used to indicate a potential matrix interference; interferences are suspected if the %D exceeds 10% and the 10x dilution result is greater than five times the Method Detection Limit (MDL). The Relative Percent Difference (RPD) for duplicate analyses is shown, and the flag "*" is used to indicate when the RPD has exceeded 20% and the quantitated results exceed the MDL. An estimate of the sample detection limit can be obtained by multiplying the analyte's "IDL" value by the appropriate sample "Dil Factor." It should be noted that the processing blank has not been subtracted from the reported sample results. Also, no CRI MDL standard was analyzed; see Deficiency Report DR-93-033.

Core 55 Composite (93-08755-H) -- Tables 2-1a and 2-1b: The ICP results for the core composite show the major analytes to be Al, Fe, and Na; totalling approximately 36%, wet weight. The comparison between the fusion results and acid digestion results are reasonably good for Fe and Na; however, Al results from the fusions are about 50% greater than for the acid digestion. The %D between the 2x and 10x dilutions for both the sample and duplicate is very good, indicating that the instrument results obtained on the fusion solutions are reliable. The RPDs for the major concentration analytes were acceptable but relatively high, indicating difficulty in obtaining representative sample for the fusion preparation (Note: fusion procedure uses only 0.2 g of material for

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dissolution which may add significantly to the apparent heterogeneity). It should be noted that the analyte concentrations for the "duplicate" are consistently 10-20% lower than the "sample" for all analytes above the MDL. The processing blank shows no analyte concentrations above the MDL and the post spike analysis shows good recovery for all analytes. The Cd recoveries on analytical QC standards demonstrated consistently high recoveries (noninally 112%); therefore, the reported core composite Cd results were presented for information only. It should be noted that the Cd results for the homogenization test samples are significantly lower. The arsenic results for this set of analyses are unusable due to a malfunction of the ICP's As channel.

Homogenization Tests: Core 55 Composite (93-08755-H Top/Bottom) -- Table 2-1c: The KOH fusion results for the core composite homogenization test shows no "statistical" difference (based on a mean Student "t") between the top and the bottom sample results; this is primarily because the analytical variability between all samples (top, top duplicate, bottom, and bottom duplicate) is very large. A significant number of the RPDs (comparing all four samples) are higher than normal (i.e., 10-60% verses <10%), indicating a very poor homogenization which is most likely due to particle size issues or sampling variability which can be attributed to the very small sample weights used for fusions preparations. Particularly disturbing are the Fe and Na results which show percent differences between samples from 32% to 55%; potentially resulting from the dark particles observed in the primarily white sample. Based on these results, an attempt was made to improve homogenization by reducing the particle size through a grinding process; however, due to the lack of sample, no further ICP homogenization tests were performed. Difficulties in obtaining quality duplicate sampling compromise the accuracy of the full suite of characterization analyses performed, as well as adversely affecting the ability to obtain good RPDs or pre-digestion spike recoveries. No sample QC was performed other than the duplicate analyses for the top and bottom samples and a fusion processing blank.

Table 2-1a: T-102 Core 55, ICP Core Composite KOH Fusion

Project: SST
Procedure: PNL-ALO-211
M&TE: JA ICP W873520

ICP ANALYSIS REPORT
** Sample Results **

Analyze Date: 08/23/93
File: m090293d
Report Page 1 of 2

	Samp Log#:	93-08755h1	93-08755h1		93-08755h2	93-08755h2		93-08755h3		
	ICP Run #:	42	41		44	43		40		
	Dil Fctr:	11062.28	55311.41		11179.91	55899.53		11117.98		
	ICP Dil:	2.00	10.00		2.00	10.00		2.00		
		<----------Sample---------->			<--------Duplicate-------->			<----Blank----->		**Calculated**
	Analyte	ug/g	ug/g	%D	ug/g	ug/g	%D	ug/g	>MDL?	IDL ug/mL	MDL ug/mL
	Ag	(919) J	(1,289)		(908)	(1,174)		(927)		0.010	0.1000
	Al	311,293	312,841	0%	287,212	295,541	3%	ND		0.060	0.6000
	As	xx	xx		xx	xx		xx		0.080	0.8000
	B	(406) R	ND		(306)	ND		(422)		0.020	0.2000
	Ba	ND	ND		ND	ND		ND		0.010	0.1000
	Be	ND	ND		ND	ND		ND		0.005	0.0500
	Bi	ND	ND		ND	ND		ND		0.500	5.0000
	Ca	(781)	ND		(697)	ND		ND		0.050	0.5000
	Cd	(258) J	(1,001)		(239)	(872)		(282)		0.005	0.0500
	Ce	ND R	ND		ND	ND		ND		0.100	1.0000
	Co	ND R	ND		ND	ND		ND		0.010	0.1000
	Cr	(806)	ND		(767)	ND		ND		0.020	0.2000
	Cu	55 R	ND		(60)	ND		(168)		0.005	0.0500
	Dy	ND R	ND		ND	ND		ND		0.050	0.5000
	Eu	ND R	ND		ND	ND		ND		0.200	2.0000
	Fe	19,813	19,470	2%	16,300	16,311	0%	(550)		0.010	0.1000
	Gd	ND R	ND		ND	ND		ND		0.500	5.0000
	K	n/a	n/a		n/a	n/a		n/a		1.000	10.0000
	La	ND R	ND		ND	ND		ND		0.050	0.5000
	Li	ND R	ND		ND	ND		ND		0.030	0.3000
	Mg	ND R	ND		ND	ND		ND		0.100	1.0000
	Mn	1,010	(1,051)		903	(928)		(96)		0.005	0.0500
	Mo	ND R	ND		ND	ND		ND		0.030	0.3000
	Na	34,238	(33,242)		28,598	(28,553)		(2,457)		0.080	0.8000
	Nd	(1,302) R	(6,621)		(1,939)	(4,779)		(652)		0.050	0.5000
	Ni	n/a	n/a		n/a	n/a		n/a		0.030	0.3000
	P	(1,621)	ND		(1,446)	ND		ND		0.100	1.0000
	Pb	(2,008) J	ND		(1,836)	ND		(1,938)		0.060	0.6000
	Pd	ND R	ND		ND	ND		ND		0.300	3.0000
	Rh	ND R	ND		ND	ND		ND		0.300	3.0000
	Ru	ND R	ND		ND	ND		ND		0.200	2.0000
	Sb	ND	ND		ND	ND		ND		0.050	0.5000
	Se	ND	ND		ND	ND		ND		0.100	1.0000
	Si	(3,534)	ND		(2,945)	ND		ND		0.080	0.8000
	Sn	ND R	ND		ND	ND		ND		1.000	10.0000
	Sr	ND R	ND		ND	ND		ND		0.005	0.0500
	Te	ND R	ND		ND	ND		ND		0.500	5.0000
	Th	ND R	ND		ND	ND		ND		0.800	8.0000
	Ti	(61) R	ND		(59)	ND		ND		0.005	0.0500
	Tl	ND R	ND		ND	ND		ND		0.500	5.0000
	U	ND	ND		ND	ND		ND		2.000	20.0000
	V	ND	ND		ND	ND		ND		0.010	0.1000
	W	ND R	ND		ND	ND		ND		0.200	2.0000
	Y	ND R	ND		ND	ND		ND		0.010	0.1000
	Zn	(926) R	ND		(678)	ND		(576)		0.020	0.2000
	Zr	ND UJ	ND		ND	ND		ND		0.010	0.1000

Note:
1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) The process "Blank" has not been subtracted from the "Sample & Duplicate" results.
4) At 50-100 times the IDL, precision is estimated at +/-10% and accuracy at +/-15%.
5) "ND"; Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr)
6) Potential chemical/physical interferences if Sample Results >5X MDL and Percent Difference (%D) >10%.
7) ">MDL?" = Yes: Blank results are above MDL and require investigation for potential contamination.
8) "xx": Arsenic channel malfunction; results erratic and unusable.
Data, including calibration/QC, archived File ICP-325-405-I-08/23/93

dw 10/25/93

Table 2-1b: T-102 Core 55, ICP Core Composite KOH Fusion - QC Results

Project: SST
Procedure:PNL-ALO-211
H&TE: JA ICP W873520

ICP ANALYSIS REPORT
**** QC Results *****

08/23/93 : Analyzed
m090293d : File

Samp Log# >>> 93-08755ps
ICP Run # >>> 51
Dil Fctr >>> 110.00
ICP Dil >>> 1.00

Analyte	Category	Average a1&a2 ug/g	RPD	20% Flg	PreSpk STD ug/mL	Spike Added ug/g	Spike+ Sample ug/g	Rec	Spk Flg	Blk Spike Control ug/mL	Rec	Spk Flg	PostSpk STD ug/mL	Post Spike ug/mL	Rec	Spk Flg
Ag	B	---			50								50	47	94%	
Al	A	299,252	8%		250								250	n/a		
As	C	xx			200								200	xx		
B	B	---														
Ba	-	---			50		No sample spike or process blank spike						50	51	102%	
Be	C	---			5		analyzed with fusion prepared samples.						5	5	108%	
Bi	B	---			500								500	522	104%	
Ca	A	---			500								500	508	102%	
Cd	B	---			25								25	24	98%	
Ce	B	---														
Co	-	---														
Cr	A	---			50								50	51	102%	
Cu	-	---														
Dy	-	---														
Eu	-	---														
Fe	A	18,056	19%		50								50	n/a		
Gd	-	---														
K	B	n/a			1000								1000	n/a		
La	B	---														
Li	B	---														
Mg	A	---														
Mn	A	957	11%		10								10	9	90%	
Mo	B	---														
Na	A	31,418	18%		1000								1000	899	90%	
Nd	B	---														
Ni	B	n/a			50								50	n/a		
P	A	---														
Pb	C	---			500								500	501	100%	
Pd	-	---														
Rh	-	---														
Ru	-	---														
Sb	C	---														
Se	C	---			500								500	623	125%	
Si	B	---			500								500	535	107%	
Sn	-	---														
Sr	B	---														
Te	-	---														
Th	-	---														
Ti	B	---														
Tl	C	---														
U	-	---			1000								1000	1069	107%	
V	-	---			50								50	50	101%	
W	-	---														
Y	-	---														
Zn	-	---														
Zr	A	---			50								50	52	103%	

Note:
1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) At 50-100 times the IDL., precision is estimated at +/-10% and accuracy at +/-15%.
3) Where results are >IDL the "Blank" have been subtracted from the "Spike Control".
4) Spike Flag (N) indicates spike is outside the QC recovery criteria.
5) 20% Flag (*): RPD > 20% and both sample and duplicate results >MDL.
6) If spike is <25% of sample concentration, Rec is not calculated as indicated by the "n/a".
7) If sample or duplicate results are <MDL then average is not calculated (i.e., "---").
8) "xx": Arsenic channel malfunction; results erratic and unusable.

Table 2-1c: T-102 Core 55, ICP Homogenization Check KOH Fusion

PROCEDURE: PNL-ALO-211 ICP ANALYSIS REPORT -- Homogenization Test Analyzed Date: 07/23/93
M&TE: JA ICP W873520 File: m072393
PROJECT: SST

		93-	93-		93-	93-		93-			
Samp Log#:		8755h-1T	8755h-2T		8755h-1B	8755h-2B		8755h-3T			
ICP Dil:		1.00	1.00		1.00	1.00		1.00			
Dil Fctr:		3653.2	3630.0		3612.0	3634.9		3632.5			
ICP Run #:		28	32		34	37		26			
	Analyte	Top ug/g	Top-Dup ug/g	20% RPD Flag	Bottom ug/g	Bot-Dup ug/g	20% RPD Flag	Blank ug/g	Blank >MDL?	**Calculated** IDL ug/mL	MDL ug/mL
J	Ag	(329)	(314)		J (306)	(309)		(313)		0.010	0.1000
	Al	284,032	279,477	2%	266,060	294,571	10%	(704)		0.060	0.6000
	As	ND	ND		ND	ND		ND		0.080	0.8000
R	B	(348)	(142)		R (216)	(300)		(293)		0.020	0.2000
	Ba	ND	ND		ND	ND		ND		0.010	0.1000
	Be	ND	ND		ND	ND		ND		0.005	0.0500
R	Bi	ND	ND		R ND	ND		ND		0.500	5.0000
J	Ca	(828)	(770)		J (733)	(716)		(351)		0.050	0.5000
J	Cd	(25)	ND		J (19)	ND		(20)		0.005	0.0500
R	Ce	ND	ND		R ND	ND		ND		0.100	1.0000
R	Co	ND	ND		R ND	ND		ND		0.010	0.1000
	Cr	735	779	6%	786	(654)		ND		0.020	0.2000
R	Cu	(49)	(37)		R (36)	(34)		(38)		0.005	0.0500
R	Dy	ND	ND		R ND	ND		ND		0.050	0.5000
R	Eu	ND	ND		R ND	ND		ND		0.200	2.0000
	Fe	16,878	16,905	0%	J 15,221	8,949	52%	(201)		0.010	0.1000
R	Gd	ND	ND		R ND	ND		ND		0.500	5.0000
	K	n/a	n/a		n/a	n/a		n/a		1.000	10.0000
R	La	ND	ND		R ND	ND		ND		0.050	0.5000
R	Li	ND	ND		R ND	ND		ND		0.030	0.3000
R	Mg	ND	ND		R ND	ND		ND		0.100	1.0000
J	Mn	705	981	33%	J 1,187	672	55%	(159)		0.005	0.0500
R	Mo	ND	ND		R ND	ND		ND		0.030	0.3000
	Na	30,160	28,590	5%	J 34,682	25,270	31%	(2,574)		0.080	0.8000
R	Nd	(389)	(198)		R (354)	ND		(332)		0.050	0.5000
	Ni	n/a	n/a		n/a	n/a		n/a		0.030	0.3000
	P	(952)	(870)		(833)	(840)		ND		0.100	1.0000
	Pb	(593)	(695)		(509)	(410)		ND		0.060	0.6000
R	Pd	ND	ND		R ND	ND		ND		0.300	3.0000
R	Rh	ND	ND		R ND	ND		ND		0.300	3.0000
R	Ru	ND	ND		R ND	ND		ND		0.200	2.0000
	Sb	ND	ND		ND	ND		ND		0.050	0.5000
	Se	ND	ND		ND	ND		ND		0.100	1.0000
J	Si	(2,647)	3,201		J (2,839)	(2,571)		(908)		0.080	0.8000
R	Sn	ND	ND		R ND	ND		ND		1.000	10.0000
R	Sr	(23)	(24)		R (25)	(21)		ND		0.005	0.0500
R	Te	ND	ND		R ND	ND		ND		0.500	5.0000
R	Th	ND	ND		R ND	ND		ND		0.800	8.0000
R	Ti	(50)	(46)		R (44)	(53)		ND		0.005	0.0500
R	Tl	ND	ND		R ND	ND		ND		0.500	5.0000
	U	ND	ND		ND	ND		ND		2.000	20.0000
	V	ND	ND		ND	ND		ND		0.010	0.1000
R	W	ND	ND		R ND	ND		ND		0.200	2.0000
R	Y	ND	ND		R ND	ND		ND		0.010	0.1000
R	Zn	(518)	916		R (332)	(363)		(270)		0.020	0.2000
UJ	Zr	ND	ND		UJ ND	ND		ND		0.010	0.1000

Note: 1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) Sample results have not been adjusted for "blank" contribution.
4) At 50-100 times the IDL, precision is estimated at +/-10% and accuracy at +/-15%.
5) "ND": Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr).
6) 20% "*" flag: RPD >20% and both sample results >MDL.
7) ">MDL" = Yes: Blank results are above MDL and require investigation for potential contamination.

Data, including calibration/QC, archived File ICP-325-405-1- 07/23/93

ASD
10-25-93

ICP Analysis -- Acid Digestion Results

ICP analyses were performed on acid digestions prepared from Core 55 composite material. The core composite samples were prepared following procedure PNL-ALO-101, "Acid Digestion for Metals Analysis" (i.e., HNO_3/HCl digestion), and analyzed following procedure PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry." The acid digestions were performed in the SAL and the digestates transferred to the Inorganic Analysis Group for subsequent ICP analysis. All ICP analyses were performed on a Jarrell-Ash ICP system with interelement corrections for spectral interferences being performed on-line.

The acid digestion results for the composite sample, duplicate, and blank are reported along with the associated sample QC results. Core 55 composite samples were analyzed at both a 2x and 10x dilution with the corresponding percent difference (%D) used to indicate a potential matrix interference; interferences are suspected if the %D exceeds 10% and the 10x dilution result is greater than five times the MDL. The RPD for duplicate analyses is shown, and the flag "*" is used to indicate when the RPD has exceeded 20% and the quantitated results exceed the MDL. An estimate of the sample detection limit can be obtained by multiplying the analyte's "IDL" value by the appropriate sample "Dil Factor." It should be noted that the processing blank has not been subtracted from the reported sample results. However, processing blank results greater than IDL are subtracted from the Blank Spike control prior to determining the percent spike recovery. Also, no CRI MDL standard was analyzed; see Deficiency Report DR-93-033.

Core 55 Composite (93-08755-A) -- Tables 2-2a through 2-2b: The acid digestion results for core composite correlate reasonably well with those from the fusion preparations for Fe & Na; however, Al and Si are significantly lower, as would be expected for the less robust acid digestion/leach. The %D between the 2x and 10x dilutions for both the sample and duplicate is very good, indicating that the instrument results obtained on the digestion solutions are reliable. The RPD values for Al, Fe, and Na analytes are acceptable (i.e., <20%); indicating adequate homogenization, sub-sampling, and analytical precision. Three analytes

(B, Ca, and Na) demonstrated blank concentrations above the MDL; the B and Ca "contamination" significantly impact the accuracy of the B and Ca reported results. The sample and duplicate results indicate that the primary analytes are Al, Fe, and Na; totalling approximately 20%, wet weight. Also of interest are the Cr and Pb results; since if moderately TCLP leachable, either analyte would classify the tank material as toxic.

The full suite of QC (i.e., duplicate, pre-spike, and post-spike) was performed. The percent recoveries for the Blank Spike "control" are reasonably good with most recoveries being between 80-120%; except Bi and K which recovered at 69% and 73%, respectively. The percent recoveries for the spiked samples are good with recoveries being within acceptable limits for most category A and B analytes for which spiking was performed. The exception is silicon which recovered at only 39%; fusion results should be used to quantitate Si. For a few spikes, recovery is meaningless since the spike is less than 25% of the sample's measured concentration. The pre-digestion spike additions for Al, Cr, Fe, Mn, and Na were at insufficient levels for recovery quantitation. All post-digestion spikes, except Si, met the 75-125% acceptance criteria. The arsenic results for this set of analyses are unusable due to a malfunction of the ICP's As channel.

Table 2-2a: T-102 Core 55, ICP Core Composite Acid Digestion

Project: SST
Procedure: PNL-ALO-211
H&TE: JA ICP W873520

ICP ANALYSIS REPORT
** Sample Results **

Analyze Date: 08/25/93
File: m082593
Report Page 1 of 2

Samp Log#:		93-08755a1	93-08755a1		93-08755a2	93-08755a2		93-08755a3				
ICP Run #:		25	24		27	26		23				
Dil Fctr:		398.96	1994.81		400.24	2001.20		399.68				
ICP Dil:		2.00	10.00		2.00	10.00		2.00			**Calculated**	
		<---------Sample--------->			<--------Duplicate-------->			<----Blank----->			IDL	MDL
	Analyte	ug/g	ug/g	%D	ug/g	ug/g	%D	ug/g	>MDL?		ug/mL	ug/mL
	Ag	(15)	(20)		(18)	(21)		ND			0.010	0.1000
	Al	145,262	149,751	3%	165,419	172,023	4%	(33)			0.060	0.6000
	As	xx	xx		xx	xx		xx			0.080	0.8000
R	B	201	(216)		153	(166)		233	Yes		0.020	0.2000
	Ba	(12)	ND		(12)	ND		ND			0.010	0.1000
	Be	ND	ND		ND	ND		ND			0.005	0.0500
R	Bi	ND	ND		ND	ND		(283)			0.500	5.0000
J	Ca	625	(689)		556	(642)		310	Yes		0.050	0.5000
	Cd	(11)	(15)		(11)	(14)		ND			0.005	0.0500
R	Ce	ND	ND		ND	ND		ND			0.100	1.0000
R	Co	ND	ND		(4)	ND		ND			0.010	0.1000
	Cr	737	772	5%	748	779	4%	ND			0.020	0.2000
R	Cu	(14)	(21)		(14)	(19)		(6)			0.005	0.0500
R	Dy	ND	ND		ND	ND		ND			0.050	0.5000
R	Eu	ND	ND		ND	ND		ND			0.200	2.0000
	Fe	19,254	20,108	4%	20,204	21,233	5%	(10)			0.010	0.1000
R	Gd	ND	ND		ND	ND		ND			0.500	5.0000
UJ	K	ND	ND		ND	ND		ND			1.000	10.0000
R	La	ND	ND		ND	ND		ND			0.050	0.5000
R	Li	ND	ND		ND	ND		ND			0.030	0.3000
R	Mg	(107)	ND		(107)	ND		(41)			0.100	1.0000
	Mn	755	783	4%	807	844	5%	ND			0.005	0.0500
R	Mo	ND	ND		ND	ND		ND			0.030	0.3000
	Na	27,221	27,947	3%	27,745	29,137	5%	462	Yes		0.080	0.8000
R	Nd	263	(374)		283	(324)		ND			0.050	0.5000
	Ni	(66)	(76)		(70)	(80)		ND			0.030	0.3000
	P	552	(614)		586	(639)		ND			0.100	1.0000
	Pb	374	(417)		421	(474)		ND			0.060	0.6000
R	Pd	ND	ND		ND	ND		ND			0.300	3.0000
R	Rh	ND	ND		ND	ND		ND			0.300	3.0000
R	Ru	ND	ND		ND	ND		ND			0.200	2.0000
	Sb	(40)	ND		(50)	ND		ND			0.050	0.5000
	Se	(63)	ND		(74)	ND		ND			0.100	1.0000
R	Si	853	(912)		820	(878)		(118)			0.080	0.8000
R	Sn	ND	ND		ND	ND		ND			1.000	10.0000
R	Sr	(17)	(18)		(18)	(19)		ND			0.005	0.0500
R	Te	ND	ND		ND	ND		ND			0.500	5.0000
R	Th	ND	ND		ND	ND		ND			0.800	8.0000
R	Ti	(9)	(10)		(11)	(12)		ND			0.005	0.0500
R	Tl	ND	ND		ND	ND		ND			0.500	5.0000
	U	ND	ND		ND	ND		ND			2.000	20.0000
	V	ND	ND		ND	ND		ND			0.010	0.1000
R	W	ND	ND		ND	ND		ND			0.200	2.0000
R	Y	(6)	ND		(6)	ND		ND			0.010	0.1000
R	Zn	98	(101)		123	(128)		ND			0.020	0.2000
J	Zr	42	(39)		44	(42)		ND			0.010	0.1000

ASD 10-27-93
R
ASD 10-25-93

Note:
1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) The process "Blank" has not been subtracted from the "Sample & Duplicate" results.
4) At 50-100 times the IDL, precision is estimated at +/-10% and accuracy at +/-15%.
5) "ND": Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr)
6) Potential chemical/physical interferences if Sample Results >5X MDL and Percent Difference (%D) >10%.
7) ">MDL?" = Yes: Blank results are above MDL and require investigation for potential contamination.
8) "xx": Arsenic channel malfunction; results erratic and unusable.
Data, including calibration/QC, archived File ICP-325-405-1-08/25/93

Table 2-2b: T-102 Core 55, ICP Core Composite Acid Digestion - QC Results

Project: SST
Procedure:PNL-ALO-211
H&TE: JA ICP W873S20

ICP ANALYSIS REPORT
**** QC Results *****

08/25/93 : Analyzed
m082593 : File
Report Page 1 of 2

					Samp Log# >>>	93-08755a4		93-08755a5			93-08755ps		
					ICP Run # >>>	32		34			35		
					Dil Fctr >>>	399.92		192.68			110.00		
					ICP Dil >>>	2.00		2.00			1.00		
Category					PreSpk	Spike	Spike+			Blk Spike			PostSpk	Post		
Analyte		Average		20%	STD	Added	Sample		Spk	Control		Spk	STD	Spike		Spk
		al&a2														
		ug/g	RPD	Flg	ug/mL	ug/g	ug/g	Rec	Flg	ug/mL	Rec	Flg	ug/mL	ug/mL	Rec	Flg
Ag	B	---			50	104	108	90%		46	92%		50	44	88%	
Al	A	155,341	13%		250	520	203,119	n/a		225	90%		250	n/a		
As	C	xx			200	415	xx			xx			200	xx		
B	B	177	27%	*			205									
Ba	-	---			50	104	112	96%		48	96%		50	48	97%	
Be	C	---			5	10	(10)	100%		5	94%		5	5	101%	
Bi	B	---			500	1038	(1,078)	104%		343	69%	N	500	509	102%	
Ca	A	591	12%		500	1038	1,567	91%		469	94%		500	434	87%	
Cd	B	---			25	52	60	95%		24	98%		25	23	91%	
Ce	B	---					ND									
Co	-	---					(5)									
Cr	A	743	1%		50	104	836	n/a		49	98%		50	n/a		
Cu	-	---					(15)									
Dy	-	---					ND									
Eu	-	---					ND									
Fe	A	19,729	5%		50	104	11,106	n/a		51	102%		50	n/a		
Gd	-	---					ND									
K	B	---			1000	2076	(1,629)	78%		728	73%	N	1000	801	80%	
La	B	---					ND									
Li	B	---					ND									
Mg	A	---					(100)									
Mn	A	781	7%		10	21	783	n/a		10	97%		10	n/a		
Mo	B	---					ND									
Na	A	27,483	2%		1000	2076	30,014	n/a		934	93%		1000	n/a		
Nd	B	273	7%				340									
Ni	B	---			50	104	174	103%		49	98%		50	43	87%	
P	A	569	6%				601									
Pb	C	398	12%		500	1038	1,344	93%		480	96%		500	441	88%	
Pd	-	---					ND									
Rh	-	---					ND									
Ru	-	---					ND									
Sb	C	---					(69)									
Se	C	---			500	1038	1,217	111%		545	109%		500	542	108%	
Si	B	837	4%		500	1038	1,259	39%	N	517	103%		500	637	127%	N
Sn	-	---					ND									
Sr	B	---					(18)									
Te	-	---					ND									
Th	-	---					ND									
Ti	B	---					(11)									
Tl	C	---					ND									
U	-	---			1000	2076	(2,477)	119%		893	89%		1000	1207	121%	
V	-	---			50	104	96	93%		45	91%		50	47	94%	
W	-	---					ND									
Y	-	---					(7)									
Zn	-	111	22%	*			92									
Zr	A	43	5%		50	104	146	100%		46	93%		50	49	97%	

Note:
1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) At 50-100 times the IDL., precision is estimated at +/-10% and accuracy at +/-15%.
3) Where results are >IDL the "Blank" have been subtracted from the "Spike Control".
4) Spike Flag (N) indicates spike is outside the QC recovery criteria.
5) 20% Flag (*): RPD > 20% and both sample and duplicate results >MDL.
6) If spike is <25% of sample concentration, Rec is not calculated as indicated by the "n/a".
7) If sample or duplicate results are <MDL then average is not calculated (i.e., "---").
8) "xx": Arsenic channel malfunction; results erratic and unusable.

ICP Analysis -- Water Leach Results

ICP analyses were performed on water leaches prepared from Core 55 composite material. The core composite samples were leached following procedure PNL-ALO-103, "Water Leach of Sludges, Soils, and Other Solid Samples," and then analyzed following procedure PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry." The water leaches were performed in the SAL and the sample aliquots transferred to the Inorganic Analysis Group for subsequent ICP analysis. All ICP analyses were performed on a Jarrell-Ash ICP system with interelement corrections for spectral interferences being performed on-line.

The water leach results for the composite sample, duplicate, and blank are reported along with the associated sample QC results. The leachates were analyzed at both a 2x and 10x dilution with the corresponding percent difference (%D) used to indicate a potential matrix interference; interferences are suspected if the %D exceeds 10% and the 10x dilution result is greater than five times the MDL. The RPD for duplicate analyses is shown, and the flag "*" is used to indicate when the RPD has exceeded 20% and the quantitated results exceed the MDL. An estimate of the sample detection limit can be obtained by multiplying the analyte's "IDL" value by the appropriate sample "Dil Factor." It should be noted that the processing blank has not been subtracted from the reported sample results. However, processing blank results greater than IDL are subtracted from the Blank Spike control prior to determining the percent spike recovery. Also, no CRI MDL standard was analyzed; see Deficiency Report DR-93-033.

Core 55 Composite (93-08755-C) -- Table 2-3a through 2-3b: The only major water soluble analyte appears to be Na, with very minor contributions from Al, Cr, Fe and P. The components measured by the ICP on the water leach account for only about three percent of the total sample wet weight. This water soluble fraction represents about a tenth of the wet weight fraction of analytes measured from the fusion preparation, which is considered to be a complete dissolution. The sample and duplicate RPD is considered very good for the Na results and adequate for the remaining analytes at low concentrations. However, we failed to meet the ±20% RPD acceptance criteria for analytes 10x the IDL (i.e., at the MDL).

The full suite of sample QC (i.e., duplicate, pre-spike, and post-spike) was performed. The percent recoveries for the Blank Spike "control" are reasonably good with all recoveries being between 80-120%; except Ca which shows a high bias (i.e., 167% recovery). The percent recoveries for the spiked sample are generally very poor with recoveries ranging from "not detectable" to well in excess of 200%. The primary explanation for this phenomenon is that the high acid spike solution has been added to the sample during the leaching process; this changes the leaching characteristics of the leach and 1) extracts higher concentrations of some analytes and 2) leads to precipitation of spiking analytes due to a significant pH change. DR-93-033 addresses the need for spiking solutions for "water soluble" analytes which can be added during the leaching process. For a few spikes, recovery is meaningless since the spike is less than 25% of the sample's measured concentration; the pre-digestion spikes additions for Cr and Na were at insufficient levels for recovery quantitation. The post-spike recoveries were all within the 75%-125% acceptance criteria, except Si which recovered slightly high at 127%. The arsenic results for this set of analyses are unusable due to a malfunction of the ICP's As channel.

Table 2-3a: T-102 Core 55, ICP Core Composite Water Leach

Project: SST
Procedure: PNL-ALO-211
H&TE: JA ICP WB73520

ICP ANALYSIS REPORT
** Sample Results **

Analyze Date: 08/23/93
File: m082393

Samp Log#:		93-08755c1	93-08755c1		93-08755c2	93-08755c2		93-08755c3			
ICP Run #:		33	32		35	34		31			
Dil Fctr:		201.58	1007.92		202.80	1014.01		206.50			
ICP Dil:		2.00	10.00		2.00	10.00		2.00		**Calculated**	
		<---Sample--->			<---Duplicate--->			<---Blank--->			
Analyte		ug/g	ug/g	%D	ug/g	ug/g	%D	ug/g	>MDL?	IDL ug/mL	MDL ug/mL
	Ag	ND	ND		ND	ND		ND		0.010	0.1000
J	Al	950	834	12%	788	(595)		ND		0.060	0.6000
	As	xx	xx		xx	xx		xx		0.080	0.8000
R	B	ND	ND		ND	ND		ND		0.020	0.2000
	Ba	ND	ND		ND	ND		ND		0.010	0.1000
	Be	ND	ND		ND	ND		ND		0.005	0.0500
R	Bi	ND	ND		ND	ND		ND		0.500	5.0000
J	Ca	(14)	ND		(13)	ND		ND		0.050	0.5000
	Cd	ND	ND		ND	ND		ND		0.005	0.0500
R	Ce	ND	ND		ND	ND		ND		0.100	1.0000
R	Co	ND	ND		ND	ND		ND		0.010	0.1000
	Cr	767	770	0%	776	774	0%	ND		0.020	0.2000
R	Cu	ND	ND		ND	ND		ND		0.005	0.0500
R	Dy	ND	ND		ND	ND		ND		0.050	0.5000
R	Eu	ND	ND		ND	ND		ND		0.200	2.0000
J	Fe	130	(92)		91	(17)		ND		0.010	0.1000
R	Gd	ND	ND		ND	ND		ND		0.500	5.0000
	K	ND	ND		ND	ND		ND		1.000	10.0000
R	La	ND	ND		ND	ND		ND		0.050	0.5000
R	Li	ND	ND		ND	ND		ND		0.030	0.3000
R	Mg	ND	ND		ND	ND		ND		0.100	1.0000
J	Mn	10	(8)		(6)	ND		ND		0.005	0.0500
R	Mo	(7)	ND		(7)	ND		ND		0.030	0.3000
	Na	28,121	28,524	1%	29,569	29,041	2%	ND		0.080	0.8000
R	Nd	ND	ND		ND	ND		ND		0.050	0.5000
	Ni	ND	ND		ND	ND		ND		0.030	0.3000
	P	408	(420)		423	(402)		ND		0.100	1.0000
	Pb	ND	ND		ND	ND		ND		0.060	0.6000
R	Pd	ND	ND		ND	ND		ND		0.300	3.0000
R	Rh	ND	ND		ND	ND		ND		0.300	3.0000
R	Ru	ND	ND		ND	ND		ND		0.200	2.0000
	Sb	ND	ND		ND	ND		ND		0.050	0.5000
	Se	ND	ND		ND	ND		ND		0.100	1.0000
R	Si	(45)	ND		(47)	ND		ND		0.080	0.8000
R	Sn	ND	ND		ND	ND		ND		1.000	10.0000
R	Sr	ND	ND		ND	ND		ND		0.005	0.0500
R	Te	ND	ND		ND	ND		ND		0.500	5.0000
R	Th	ND	ND		ND	ND		ND		0.800	8.0000
R	Ti	ND	ND		ND	ND		ND		0.005	0.0500
R	Tl	ND	ND		ND	ND		ND		0.500	5.0000
	U	ND	ND		ND	ND		ND		2.000	20.0000
J	V	ND	ND		ND	ND		ND		0.010	0.1000
R	W	ND	ND		ND	ND		ND		0.200	2.0000
R	Y	ND	ND		ND	ND		ND		0.010	0.1000
R	Zn	ND	ND		ND	ND		ND		0.020	0.2000
J	Zr	ND	ND		ND	ND		ND		0.010	0.1000

Note: 1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) The process "Blank" has not been subtracted from the "Sample & Duplicate" results.
4) At 50-100 times the IDL, precision is estimated at +/-10% and accuracy at +/-15%.
5) "ND": Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr)
6) Potential chemical/physical interferences if Sample Results >5X MDL and Percent Difference (%D) >10%.
7) ">MDL?" = Yes: Blank results are above MDL and require investigation for potential contamination.
8) "xx": Arsenic channel malfunction; results erratic and unusable.
Data, including calibration/QC, archived File ICP-325-405-1- 08/23/93

ASD 10-25-93

Table 2-3b: T-102 Core 55, ICP Core Composite Water Leach - QC Results

Project: SST
Procedure:PNL-ALO-211
M&TE: JA ICP WB73520

ICP ANALYSIS REPORT
**** QC Results *****

08/23/93 : Analyzed
m082393 : File

Analyte	Category	Average a1&a2 ug/g	20% RPD Flg	PreSpk STD ug/mL	Spike Added ug/g	Spike+ Sample ug/g	Rec	Spk Flg	Blk Spike Control ug/mL	Rec	Spk Flg	PostSpk STD ug/mL	Post Spike ug/mL	Rec	Spk Flg
				Samp Log# >>>		93-08755c4			93-08755c5				93-08755ps		
				ICP Run # >>>		44			46				51		
				Dil Fctr >>>		192.97			203.63				110.00		
				ICP Dil >>>		2.00			2.00				1.00		
Ag	B	---		50	47	54	113%		50	101%		50	51	101%	
Al	A	869	19%	250	238	3,350	1010%	N	253	101%		250	n/a		
As	C	xx				xx						200	xx		
B	B	---				(18)									
Ba	-	---		50	47	56	118%		54	107%		50	53	106%	
Be	C	---		5	5	(5)	107%		5	105%		5	5	105%	
Bi	B	---		500	474	ND	0%	N	495	99%		500	510	102%	
Ca	A	---		500	474	1,047	218%	N	837	167%	N	500	502	100%	
Cd	B	---		25	24	29	123%		27	107%		25	26	105%	
Ce	B	---				ND									
Co	-	---				ND									
Cr	A	772	1%	50	47	745	n/a		54	108%		50	n/a		
Cu	-	---				(3)									
Dy	-	---				ND									
Eu	-	---				ND									
Fe	A	110	35% *	50	47	146	34%	N	54	107%		50	57	114%	
Gd	-	---				ND									
K	B	---		1000	948	(974)	103%		964	96%		1000	1073	107%	
La	B	---				(12)									
Li	B	---				ND									
Mg	A	---				(76)									
Mn	A	---		10	9	472	4869%	N	11	110%		10	11	111%	
Mo	B	---				ND									
Na	A	28,845	5%	1000	948	29,023	n/a		1193	119%		1000	n/a		
Nd	B	---				241									
Ni	B	---		50	47	62	131%	N	53	107%		50	55	109%	
P	A	415	3%			262									
Pb	C	---		500	474	678	143%		532	106%		500	526	105%	
Pd	-	---				ND									
Rh	-	---				ND									
Ru	-	---				ND									
Sb	C	---				ND									
Se	C	---				(22)						500	634	127%	N
Si	B	---		500	474	1,024	206%	N	580	116%		500	596	119%	
Sn	-	---				ND									
Sr	B	---				14									
Te	-	---				ND									
Th	-	---				ND									
Ti	B	---				ND									
Tl	C	---				ND									
U	-	---		1000	948	(1,622)	171%		1018	102%		1000	1243	124%	
V	-	---		50	47	31	65%		51	101%		50	51	102%	
W	-	---				ND									
Y	-	---				(4)									
Zn	-	---				(25)									
Zr	A	---		50	47	ND	0%	N	52	104%		50	53	106%	

Note:
1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) At 50-100 times the IDL., precision is estimated at +/-10% and accuracy at +/-15%.
3) Where results are >IDL the "Blank" have been subtracted from the "Spike Control".
4) Spike Flag (N) indicates spike is outside the QC recovery criteria.
5) 20% Flag (*): RPD > 20% and both sample and duplicate results >MDL.
6) If spike is <25% of sample concentration, Rec is not calculated as indicated by the "n/a".
7) If sample or duplicate results are <MDL then average is not calculated (i.e., "---").
8) "xx": Arsenic channel malfunction; results erratic and unusable.

ICP Analysis -- Acidified "Blank" Results

ICP analyses were performed on water blanks (i.e., T-102 Field Blank, HLRF Hot Cell Blank, and HLRF DIW) associated with the processing of T-102 Core 55. The water samples were acidified with HNO_3 and then analyzed following procedure PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry." The acidifications were performed by SAL staff and the sample aliquots transferred to the Inorganic Analysis Group for subsequent ICP analysis. All ICP analyses were performed on a Jarrell-Ash ICP system with interelement corrections for spectral interferences being performed on-line.

The water blanks were processed and analyzed as a single batch; the results are reported along with the associated sample QC results. All samples were analyzed at 1x and since no analytes except Na were found above 5xMDL, no serial dilutions were performed. The RPD for the duplicate analyses is shown, and the flag "*" is used to indicate when the RPD has exceeded 20% and the quantitated results exceed the MDL. An estimate of the sample detection limit can be obtained by multiplying the analyte's "IDL" value by the appropriate sample "Dil Factor"; note that at 1x the sample detection limit is the IDL. Since these samples are "blanks" and are analyzed directly, no processing blank has been analyzed with these samples. It should be noted that the arsenic results for all analyses associated with the water blanks are unusable due to a malfunction of the ICP's As channel. Also, no CRI MDL standard was analyzed; see Deficiency Report DR-93-033.

HLRF Hot Cell Blank (93-09774-R) -- Table 2-4a through 2-4b: The only analytes detected above the MDL are Na and Ca, being about 5 and 0.5 μg/mL, respectively. The RPDs for Na and Ca are very good, as would be expected for duplicate water analyses. The Cd recoveries on analytical QC standards demonstrated consistently high recoveries (nominally 112%); therefore, the reported Cd results are presented for information only. Spikes added to the sample by SAL demonstrated excellent recovery, ranging from 92% to 110%. It should be noted that only Part A of the standard spiking solution was added to the HLRF Hot Cell Blank; therefore, As and Se are not present.

HLRF DIW (93-09804-R) -- Table 2-4c: There were no analytes detected above the MDL; therefore, the DIW used for the HLRF Hot Cell Blank should not contribute significantly to the Na and Ca concentrations observed in the Hot Cell Blank.

T-102 Field Blank (93-05874-R) -- Table 2-4d: The only analytes detected above the MDL are B, Ca, Na, and Si. The Ca and Na concentrations are similar to those found in the HLRF Hot Cell Blank. The B and Si appear to be unique to the Field Blank (i.e., relative to other water blanks analyzed). The RPDs for the analytes above the MDL are excellent, which is typical for duplicate analyses of water containing low analyte concentrations.

Table 2-4a: T-102 HLRF Hot Cell Blank, Acidified

Project: SST
Procedure: PNL-ALO-211
M&TE: JA ICP WB73520

ICP ANALYSIS REPORT
** Sample Results **

Analyze Date: 09/02/93
File: m090293a
Report Page 1 of 2

Samp Log#:	93-09774r1			93-09774r2					
ICP Run #:	26			27					
Dil Fctr:	1.00			1.00					
ICP Dil:	1.00			1.00					
	<-Sample->			<-Duplicate->			<-Blank->	**Calculated**	
Analyte	ug/mL	ug/mL	XD	ug/mL	ug/mL	XD	ug/mL	IDL ug/mL	MDL ug/mL
Ag	ND	UJ		ND				0.010	0.1000
Al	ND			ND			No Blank	0.060	0.6000
As	xx			xx			Analyzed	0.080	0.8000
B	ND	R		ND				0.020	0.2000
Ba	ND			ND				0.010	0.1000
Be	ND			ND				0.005	0.0500
Bi	ND			ND				0.500	5.0000
Ca	0.513			0.508				0.050	0.5000
Cd	(0.009)	J		(0.007)				0.005	0.0500
Ce	ND	R		ND				0.100	1.0000
Co	ND	R		ND				0.010	0.1000
Cr	ND			ND				0.020	0.2000
Cu	(0.015)	R		(0.015)				0.005	0.0500
Dy	ND	R		ND				0.050	0.5000
Eu	ND	R		ND				0.200	2.0000
Fe	(0.038)			(0.039)				0.010	0.1000
Gd	ND	R		ND				0.500	5.0000
K	ND			ND				1.000	10.0000
La	ND	R		ND				0.050	0.5000
Li	ND	R		ND				0.030	0.3000
Mg	ND	R		ND				0.100	1.0000
Mn	(0.028)			(0.028)				0.005	0.0500
Mo	ND	R		ND				0.030	0.3000
Na	5.212			5.183				0.080	0.8000
Nd	(0.125)	R		(0.117)				0.050	0.5000
Ni	(0.049)			(0.048)				0.030	0.3000
P	(0.139)			(0.140)				0.100	1.0000
Pb	ND			ND				0.060	0.6000
Pd	ND	R		ND				0.300	3.0000
Rh	ND	R		ND				0.300	3.0000
Ru	ND	R		ND				0.200	2.0000
Sb	ND			ND				0.050	0.5000
Se	ND			ND				0.100	1.0000
Si	(0.143)			(0.145)				0.080	0.8000
Sn	ND	R		ND				1.000	10.0000
Sr	ND	R		ND				0.005	0.0500
Te	ND	R		ND				0.500	5.0000
Th	ND	R		ND				0.800	8.0000
Ti	ND	R		ND				0.005	0.0500
Tl	ND	R		ND				0.500	5.0000
U	ND			ND				2.000	20.0000
V	ND			ND				0.010	0.1000
W	ND	R		ND				0.200	2.0000
Y	ND	R		ND				0.010	0.1000
Zn	(0.060)	R		(0.058)				0.020	0.2000
Zr	ND	UJ		ND				0.010	0.1000

Note:
1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) The process "Blank" has not been subtracted from the "Sample & Duplicate" results.
4) At 50-100 times the IDL, precision is estimated at +/-10% and accuracy at +/-15%.
5) "ND"; Estimated Sample Detection Limit (ug/mL) = (IDL in ug/mL) * (Dil Fctr)
6) Potential chemical/physical interferences if Sample Results >5X MDL and Percent Difference (%D) >10%.
7) ">MDL?" = Yes: Blank results are above MDL and require investigation for potential contamination.
8) "xx": Arsenic channel malfunction; results erratic and unusable.
Data, including calibration/QC, archived File ICP-325-405-1-09/02/93

dlw 10/25/93

/A-45

Table 2-4b: T-102 HLRF Hot Cell Blank, Acidified - QC Results

Project: SST
Procedure:PHL-ALO-211
H&TE: JA ICP WB73520

ICP ANALYSIS REPORT
**** QC Results *****

09/02/93 : Analyzed
m090293a : File
Report Page 1 of 2

Samp Log# >>> 93-09774r4
ICP Run # >>> 28
Dil Fctr >>> 1.00
ICP Dil >>> 1.00

Analyte	Category	Average a1&a2 ug/mL	RPD	20% Flg	PreSpk STD ug/mL	Spike Added ug/mL	Spike+ Sample ug/mL	Rec	Spk Flg	Blk Spike Control ug/mL	Rec	Spk Flg	PostSpk STD ug/mL	Post Spike ug/mL	Rec	Spk Flg
Ag	B	---			50	0.492	0.456	93%					50			
Al	A	---			250	2.465	2.436	99%					250			
As	C	xx					xx						200			
B	B	---					ND							Post spike performed		
Ba	-	---			50	0.492	0.515	105%					50	with 93-08755h		
Be	C	---			5	0.049	0.052	106%					5	fusion samples		
Bi	B	---			500	4.921	5.114	104%					500			
Ca	A	0.511		1%	500	4.921	5.555	102%					500			
Cd	B	---			25	0.246	0.273	107%					25			
Ce	B	---					ND									
Co	-	---					0.010									
Cr	A	---			50	0.492	0.528	107%					50			
Cu	-	---					(0.016)									
Dy	-	---					ND									
Eu	-	---					ND									
Fe	A	---			50	0.492	0.555	105%					50			
Gd	-	---					ND									
K	B	---			1000	9.842	10.900	111%					1000			
La	B	---					ND									
Li	B	---					ND									
Mg	A	---					ND									
Mn	A	---			10	0.098	0.135	109%					10			
Mo	B	---					ND									
Na	A	5.198		1%	1000	9.842	14.880	98%					1000			
Nd	B	---					(0.376)									
Ni	B	---			50	0.492	0.574	107%					50			
P	A	---					(0.174)									
Pb	C	---			500	4.921	5.326	108%					500			
Pd	-	---					ND									
Rh	-	---					ND									
Ru	-	---					ND									
Sb	C	---					ND									
Se	C	---					(0.557)						500			
Si	B	---			500	4.921	5.563	110%					500			
Sn	-	---					ND									
Sr	B	---					ND									
Te	-	---					ND									
Th	-	---					ND									
Ti	B	---					ND									
Tl	C	---					ND									
U	-	---			1000	9.842	(10.600)	108%					1000			
V	-	---			50	0.492	0.501	102%					50			
W	-	---					ND									
Y	-	---					ND									
Zn	-	---					(0.057)									
Zr	A	---			50	0.492	0.504	102%					50			

Note:
1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) At 50-100 times the IDL., precision is estimated at +/-10% and accuracy at +/-15%.
3) Where results are >IDL the "Blank" have been subtracted from the "Spike Control".
4) Spike Flag (N) indicates spike is outside the QC recovery criteria.
5) 20% Flag (*): RPD > 20% and both sample and duplicate results >MDL.
6) If spike is <25% of sample concentration, Rec is not calculated as indicated by the "n/a".
7) If sample or duplicate results are <MDL then average is not calculated (i.e., "---").
8) "xx": Arsenic channel malfunction; results erratic and unusable.

Table 2-4c: T-102 HLRF DIW, Acidified

Project: SST
Procedure: PNL-ALO-211
H&TE: JA ICP WB73520

ICP ANALYSIS REPORT
** Sample Results **

Analyze Date: 09/02/93
File: m090293b

										Calculated	
Samp Log#:	93-09804r1			93-09804r2							
ICP Run #:	26			27							
Dil Fctr:	1.00			1.00							
ICP Dil:	1.00			1.00							
	<-----Sample----->			<-----Duplicate----->			Average		20%	IDL	MDL
Analyte	ug/mL	ug/mL	%D	ug/mL	ug/mL	%D	ug/mL	RPD	Flg	ug/mL	ug/mL
Ag	ND	UJ		ND			---			0.010	0.1000
Al	ND			ND			---			0.060	0.6000
As	xx			xx			xx			0.080	0.8000
B	ND	R		ND			---			0.020	0.2000
Ba	ND			ND			---			0.010	0.1000
Be	ND			ND			---			0.005	0.0500
Bi	ND			ND			---			0.500	5.0000
Ca	ND			ND			---			0.050	0.5000
Cd	ND	UJ		ND			---			0.005	0.0500
Ce	ND	R		ND			---			0.100	1.0000
Co	ND	R		ND			---			0.010	0.1000
Cr	ND			ND			---			0.020	0.2000
Cu	(0.017)	R		ND			---			0.005	0.0500
Dy	ND	R		ND			---			0.050	0.5000
Eu	ND	R		ND			---			0.200	2.0000
Fe	(0.013)			(0.014)			---			0.010	0.1000
Gd	ND	R		ND			---			0.500	5.0000
K	(1.192)			(1.040)			---			1.000	10.0000
La	ND	R		ND			---			0.050	0.5000
Li	ND	R		ND			---			0.030	0.3000
Mg	ND	R		ND			---			0.100	1.0000
Mn	(0.007)			(0.007)			---			0.005	0.0500
Mo	ND	R		ND			---			0.030	0.3000
Na	ND			ND			---			0.080	0.8000
Nd	(0.190)	R		(0.200)			---			0.050	0.5000
Ni	ND			ND			---			0.030	0.3000
P	ND			ND			---			0.100	1.0000
Pb	ND			ND			---			0.060	0.6000
Pd	ND	R		(0.377)			---			0.300	3.0000
Rh	ND	R		ND			---			0.300	3.0000
Ru	ND	R		ND			---			0.200	2.0000
Sb	ND			ND			---			0.050	0.5000
Se	ND			ND			---			0.100	1.0000
Si	(0.108)			(0.117)			---			0.080	0.8000
Sn	ND	R		ND			---			1.000	10.0000
Sr	ND	R		ND			---			0.005	0.0500
Te	ND	R		ND			---			0.500	5.0000
Th	ND	R		ND			---			0.800	8.0000
Ti	ND	R		ND			---			0.005	0.0500
Tl	ND	R		ND			---			0.500	5.0000
U	ND			ND			---			2.000	20.0000
V	ND			ND			---			0.010	0.1000
W	ND	R		ND			---			0.200	2.0000
Y	ND	R		ND			---			0.010	0.1000
Zn	(0.119)	R		(0.118)			---			0.020	0.2000
Zr	ND	UJ		ND			---			0.010	0.1000

Note:
1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) The process "Blank" has not been subtracted from the "Sample & Duplicate" results.
4) At 50-100 times the IDL, precision is estimated at +/-10% and accuracy at +/-15%.
5) "ND"; Estimated Sample Detection Limit (ug/mL) = (IDL in ug/mL) * (Dil Fctr)
6) Potential chemical/physical interferences if Sample Results >5X MDL and Percent Difference (%D) >10%.
7) 20% Flag (*): RPD > 20% and both sample and duplicate results >MDL.
8) "xx": Arsenic channel malfunction; results erratic and unusable.
Data, including calibration/QC, archived File ICP-325-405+09/02/93

dw 10/25/93

Table 2-4d: T-102 Field Blank, Acidified

Project: SST
Procedure: PNL-ALO-211
M&TE: JA ICP WB73520

ICP ANALYSIS REPORT
** Sample Results **

Analyze Date: 09/02/93
File: m090293c
Report Page 1 of 1

Samp Log#:	93-05874r1	93-05874r2
ICP Run #:	29	30
Dil Fctr:	1.00	1.00
ICP Dil:	1.00	1.00

Analyte	Sample ug/mL		Sample %D	Duplicate ug/mL	Duplicate ug/mL	Duplicate %D	Average ug/mL	RPD	20% Flg	**Calculated** IDL ug/mL	MDL ug/mL
Ag	ND	UJ		ND			---			0.010	0.1000
Al	ND			ND			---			0.060	0.6000
As	xx			xx			xx			0.080	0.8000
B	0.268	R		0.271			0.269	1%		0.020	0.2000
Ba	ND			ND			---			0.010	0.1000
Be	ND			ND			---			0.005	0.0500
Bi	ND			ND			---			0.500	5.0000
Ca	0.510			0.514			0.512	1%		0.050	0.5000
Cd	ND	UJ		ND			---			0.005	0.0500
Ce	ND	R		ND			---			0.100	1.0000
Co	ND	R		ND			---			0.010	0.1000
Cr	ND			ND			---			0.020	0.2000
Cu	ND	R		ND			---			0.005	0.0500
Dy	ND	R		ND			---			0.050	0.5000
Eu	ND	R		ND			---			0.200	2.0000
Fe	ND			ND			---			0.010	0.1000
Gd	ND	R		ND			---			0.500	5.0000
K	ND			ND			---			1.000	10.0000
La	ND	R		ND			---			0.050	0.5000
Li	ND	R		ND			---			0.030	0.3000
Mg	ND	R		ND			---			0.100	1.0000
Mn	(0.012)			(0.012)			---			0.005	0.0500
Mo	ND	R		ND			---			0.030	0.3000
Na	2.277			2.286			2.282	0%		0.080	0.8000
Nd	(0.170)	R		(0.213)			---			0.050	0.5000
Ni	ND			ND			---			0.030	0.3000
P	ND			ND			---			0.100	1.0000
Pb	ND			ND			---			0.060	0.6000
Pd	ND	R		ND			---			0.300	3.0000
Rh	ND	R		ND			---			0.300	3.0000
Ru	ND	R		ND			---			0.200	2.0000
Sb	ND			ND			---			0.050	0.5000
Se	ND			ND			---			0.100	1.0000
Si	1.964			1.962			1.963	0%		0.080	0.8000
Sn	ND	R		ND			---			1.000	10.0000
Sr	ND	R		ND			---			0.005	0.0500
Te	ND	R		ND			---			0.500	5.0000
Th	ND	R		ND			---			0.800	8.0000
Ti	ND	R		ND			---			0.005	0.0500
Tl	ND	R		ND			---			0.500	5.0000
U	ND			ND			---			2.000	20.0000
V	ND			ND			---			0.010	0.1000
W	ND	R		ND			---			0.200	2.0000
Y	ND	R		ND			---			0.010	0.1000
Zn	(0.037)	R		(0.036)			---			0.020	0.2000
Zr	ND	UJ		ND			---			0.010	0.1000

Note:
1) Values reliable to 2 1/2 significant digits. "( )" results are <MDL but =>IDL.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) The process "Blank" has not been subtracted from the "Sample & Duplicate" results.
4) At 50-100 times the IDL, precision is estimated at +/-10% and accuracy at +/-15%.
5) "ND": Estimated Sample Detection Limit (ug/mL) = (IDL in ug/mL) * (Dil Fctr)
6) Potential chemical/physical interferences if Sample Results >5X MDL and Percent Difference (%D) >10%.
7) 20% Flag (*): RPD > 20% and both sample and duplicate results >MDL.
8) "xx": Arsenic channel malfunction; results erratic and unusable.
Data, including calibration/QC, archived File ICP-325-405-4-09/02/93

dw 10/25/93

IA-48

Ion Chromatographic Analysis Results: Table 2-5a through 2-5d

The IC analyses were performed on water leaches prepared from Core 55 composite material and on "water blank" samples (i.e., T-102 Field Blank, HLRF Hot Cell Blank, and HLRF DIW Blank). The core composite samples were leached following procedure PNL-ALO-103, "Water Leach of Sludges, Soils, and Other Solid Samples," and then analyzed for the anions fluoride, chloride, nitrite, nitrate, phosphate, and sulfate following procedure PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography." The water blanks were analyzed directly by procedure PNL-ALO-212 after filtering. The water leaches were performed in the SAL and the sample aliquots, along with the filtered water blanks, transferred to the Inorganic Analysis Group for subsequent IC analyses. All IC analyses were performed on a Dionex 4500i Ion Chromatograph system and the lowest calibration standard for each analyte is defined as the method detection limit (MDL), the IDL has been shown to be approximately 1/2 the MDL. A CRA MDL Standard was analyzed and recovered all anions within the acceptance limit of 70% to 130%.

Core 55 Composite (93-08755-G) -- Table 2-5a: Nitrite/nitrate comprise over 90% of the anion concentration measured. For all anions, the RPD values were less than 10%, indicating consistent aliquoting leaching and very good analytical precision. The Spike Blank control demonstrated a consistently high recovery; nominally 125% for all anions. Since other QC spikes (e.g., 93-08755-C4 and 93-09774-Q4) were acceptable, the high recovery is attributed to a small spiking error and is not considered as indicative of a bias in the results.

T-102 Field Blank (93-05874-Q), HLRF Hot Cell Blank (93-09774-Q), and HLRF DIW "Blank" (93-09804-Q) -- Tables 2-5b through 2-5d: None of the water blanks contained any appreciable quantity of anions. The IC sample aliquot for the Field Blank duplicate measured nitrate at 4.4 μg/mL and appears to have been contaminated since neither the sample nor spiked sample indicate comparable nitrate levels; also, the ICP aliquot for cations shows no contamination. Since the detected anion concentrations are less than 10xIDL, the RPDs have not been calculated (with the exception of the Field Blank nitrate results). The matrix spike addition performed on the Hot Cell Blank sample, recovered all anions between 83% and 105%.

Table 2-5a: T-102 Core 55, Inorganic Analysis Summary

Analyte	ALO Log #	(1) Sample[ac] (µg/g)	(2) Duplicate[ac] (µg/g)	RPD[b] (%)	(3) Blank[ac] (µg/g)	(4) Sample[cde] Spike (%Rec)	(5) Blank[cde] Spike (%Rec)
Hg-CVAA	93-08755-D	7.7	4.8	46	(0.01)	n/a	102
Cr(VI)	93-08755-C	741	745	1	<100	p 106	--
pH	93-08755-M	9.8	9.8	0	7.5	--	--
OH	93-08755-M	ND	ND	n/a	ND	--	--
CN	93-08755-G	3.9	4.4	13	ND	80	88
NH_3-N	93-08755-C	(27)	ND	n/a	ND	p 100	--
TOC-Direct	93-08755-J	520	680	28	xx	60 & 106	--
TOC-Leach	93-08755-C	650	660	2	80	--	--
TIC-Direct	93-08755-J	2280	2660	15	xx	86 & 117	--
TIC-Leach	93-08755-C	3500	3410	3	ND	p 111	--
TC-Direct	93-08755-J	2800	3350	18	xx	--	--
TC-Leach	93-08755-C	4150	3980	4	80	p 102	--
Fluoride	93-08755-C	220	220	0	<40	94	130
Chloride	93-08755-C	300	300	0	<40	80	120
Nitrite	93-08755-C	8000	8000	0	<80	93	117
Nitrate	93-08755-C	34000	36000	6	<80	103	131
Phosphate	93-08755-C	1110	1150	4	<80	96	126
Sulfate	93-08755-C	1620	1520	6	<80	101	123

a) Results in ( ) are <MDL but >=IDL. ND = Not Detected (i.e., <IDL). "<" Result = Less Than Stated MDL.
b) RPD = "n/a" when either sample and/or duplicate are <MDL.
c) "xx": Analysis not performed on sample; e.g., only one Blank, Sample Spike, and Blank Spike analyzed per batch.
d) "n/a": Sample result >4X spiking level :::: Prefix "p" indicates "post" spike result for Sample.
e) "--": No Sample Spike or Blank Spike required for pH, OH, Cr(VI), NH_3-N, or TOC.

Table 2-5b: T-102 HLRF Hot Cell Blank, Inorganic Analysis Summary

Analyte	ALO Log #	(1) Sample[ac] (µg/mL)	(2) Duplicate[ac] (µg/mL)	RPD[b] (%)	(3) Blank[ac] (µg/mL)	(4) Sample[cde] Spike (%Rec)	(5) Blank[cde] Spike (%Rec)
pH	93-09774-P	8.4	8.5	1	xx	--	--
OH	93-09774-P	ND	ND	n/a	--	--	--
CN	93-09774-P	(0.011)[f]	(0.010)	n/a	--	124	xx
NH_3-N	93-09774-R	ND	ND	n/a	--	p 90	--
TOC-Direct	93-09774-P	3	ND	n/a	--	103	--
TIC-Direct	93-09774-P	ND	ND	n/a	--	98	--
TC-Direct	93-09774-P	3	ND	n/a	--	--	--
Fluoride	93-09774-Q	<0.25	<0.25	n/a	--	102	--
Chloride	93-09774-Q	1.0	1.1	n/a	--	94	--
Nitrite	93-09774-Q	<0.5	<0.5	n/a	--	83	--
Nitrate	93-09774-Q	1.1	1.1	n/a	--	105	--
Phosphate	93-09774-Q	<0.5	<0.5	n/a	--	91	--
Sulfate	93-09774-Q	1.9	1.8	n/a	--	92	--

a) Results in ( ) are <MDL but >=IDL. ND = Not Detected (i.e., <IDL). "<" Result = Less Than Stated MDL.
b) RPD = "n/a" when either sample and/or duplicate are <MDL.
c) "xx": Analysis not performed on sample; e.g., only one Blank, Sample Spike, and Blank Spike analyzed per batch.
d) "n/a": Sample result >4X spiking level :::: Prefix "p" indicates "post" spike result for Sample.
e) "--": No Sample Spike, Blank Spike, and/or Blank required for pH, OH, NH_3-N, CN, or TOC.
f) Analysis performed on undistilled sample; see narrative.

/A-51

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Table 2-5c: T-102 HLRF DIW Blank, Inorganic Analysis Summary

Analyte	ALO Log #	(1) Sample[ac] (µg/g)	(2) Duplicate[ac] (µg/g)	RPD[b] (%)	(3) Blank[ac] (µg/g)	(4) Sample[cde] Spike (%Rec)	(5) Blank[cde] Spike (%Rec)
pH	93-09804-P	7.9	7.9	0	xx	--	--
OH	93-09804-P	ND	ND	n/a	--	--	--
CN	93-09804-P	ND	ND[f]	n/a	--	xx	xx
NH_3-N	93-09804-R	ND	ND	n/a	--	p 90	--
TOC-Direct	93-09804-P	9	ND	n/a	--	xx	--
TIC-Direct	93-09804-P	3	7	n/a	--	xx	--
TC-Direct	93-09804-P	11	7	n/a	--	--	--
Fluoride	93-09804-Q	0.5	0.3	n/a	--	xx	--
Chloride	93-09804-Q	<0.25	<0.25	n/a	--	xx	--
Nitrite	93-09804-Q	<0.5	<0.5	n/a	--	xx	--
Nitrate	93-09804-Q	<0.5	<0.5	n/a	--	xx	--
Phosphate	93-09804-Q	<0.5	<0.5	n/a	--	xx	--
Sulfate	93-09804-Q	0.9	0.8	n/a	--	xx	--

a) Results in ( ) are <MDL but >=IDL. ND = Not Detected (i.e., <IDL). "<" Result = Less Than Stated MDL.
b) RPD = "n/a" when either sample and/or duplicate are <MDL.
c) "xx": Analysis not performed on sample; e.g., only one Blank, Sample Spike, and Blank Spike analyzed per batch.
d) "n/a": Sample result >4X spiking level :::: Prefix "p" indicates "post" spike result for Sample.
e) "--": No Sample Spike, Blank Spike, and/or Blank required for pH, OH, NH_3-N, CN, or TOC.
f) Analysis performed on undistilled sample; see narrative.

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Table 2-5d: T-102 Field Blank, Inorganic Analysis Summary

Analyte	ALO Log #	(1) Sample[ac] (µg/mL)	(2) Duplicate[ac] (µg/mL)	RPD[b] (%)	(3) Blank[ac] (µg/mL)	(4) Sample[cde] Spike (%Rec)	(5) Blank[cde] Spike (%Rec)
pH	93-05874-P	8.5	8.5	0	xx	--	--
OH	93-05874-P	ND	ND	n/a	--	--	--
CN	93-05874-P	ND	ND[f]	n/a	--	104	102
NH_3-N	93-05874-R	ND	ND	n/a	--	p 90	--
TOC-Direct	93-05874-P	11	11	n/a	--	xx	--
TIC-Direct	93-05874-P	6	4	n/a	--	xx	--
TC-Direct	93-05874-P	18	15	n/a	--	--	--
Fluoride	93-05874-Q	<0.25	<0.25	n/a	--	xx	--
Chloride	93-05874-Q	0.3	0.8	n/a	--	xx	--
Nitrite	93-05874-Q	<0.5	0.9	n/a	--	xx	--
Nitrate	93-05874-Q	0.7	4.4	145	--	xx	--
Phosphate	93-05874-Q	<0.5	0.7	n/a	--	xx	--
Sulfate	93-05874-Q	0.7	0.9	n/a	--	xx	--

a) Results in ( ) are <MDL but >=IDL. ND = Not Detected (i.e., <IDL). "<" Result = Less Than Stated MDL.
b) RPD = "n/a" when either sample and/or duplicate are <MDL.
c) "xx": Analysis not performed on sample; e.g., only one Blank, Sample Spike, and Blank Spike analyzed per batch.
d) "n/a": Sample result >4X spiking level :::: Prefix "p" indicates "post" spike result for Sample.
e) "--": No Sample Spike, Blank Spike, and/or Blank required for pH, OH, NH_3-N, CN, or TOC.
f) Analysis performed on undistilled sample; see narrative.

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Mercury by Cold Vapor Atomic Absorption Results: Table 2-5a

The Core 55 samples were digested and then analyzed by cold vapor atomic absorption (CVAA) for mercury following a modification of procedure PNL-ALO-213, "Mercury in Water, Solids, and Sludges by Manual Cold Vapor Technique." The modification has been documented by an instruction worksheet to the SAL operation and the Inorganic Analysis Group and changes the sample size, digestion volume, and heating method (i.e., from water bath to aluminum heating block). All core and QC sample digestions were performed in the SAL and the digestates transferred to the Inorganic Analysis Group for subsequent CVAA analysis. Since only limited quantities of samples can be digested within the SAL at one time, the instrument calibration standards and calibration verification standards were digested by the Inorganic Analysis Group outside the SAL. This deviation is not expected to adversely affect the reported results since the independent QC samples digested in the SAL verify the preparation and calibration. Analytical results in Appendix D6 represent analysis of all the samples on two separate days; only the result from the August 25, 1993 run which contain a valid blank spike analysis are reported in the summary table. Also, it should be noted that no CRA MDL check standard has been analyzed; see Deficiency Report DR-93-033.

The RPD for the sample and duplicate of 46% (considerably outside the 20% target criteria) indicates significant sample inhomogeneity (with respect to Hg) within the composite. At 5-8 μg/g, the mercury concentration is moderately high; however, nearly all the mercury has to be TCLP leachable for the core material to be classified as toxic based on the mercury concentration. The Spike Blank "control" was recovered at 102%, indicating that the preparation and analysis operations were good. The spiked sample recovery was not recoverable since the concentration of the mercury in the samples was significantly higher than the spiking level. Assuming a sample weight of approximately 0.5 g, the IDL and MDL are 0.002 μg/g and 0.02 μg/g, respectively.

Chromium (VI) Results: Table 2-5a

The Chromium (VI) analyses were performed on water leaches prepared from Core 55 composite material. The core composite samples were leached following procedure PNL-ALO-103, "Water Leach of Sludges, Soils, and Other Solid Samples," and then analyzed following procedure PNL-ALO-227, "Determination of Cr(VI) in Aqueous Samples." The water leaches were conducted in the SAL and sample aliquots then transferred to the Inorganic Analysis Group for subsequent Cr(VI) analysis. Other than the analysis of duplicates and a blank, no other QC (e.g., leach spikes) was performed. However, a post-leach analytical spike was recovered at 106%, indicating that Cr(VI) is compatible with the leachate. At 1%, the RPD for the sample and duplicate is very good. The total Cr analysis by ICP (Table 2-3a) is in excellent agreement with the measured Cr(VI); with the Cr by ICP being about 770 μg/g and the Cr(VI) being about 740 μg/g. The MDL for the method is 2 μg Cr; this results in a reported MDL of 100 μg/g for the sample size and leach volume used. It should be noted that the analysis protocol established by the QAPjP was not followed; this is documented in Deficiency Report DR-93-041.

OH^- and pH Results: Table 2-5a through 2-5d

The pH and OH^- analyses were performed on water leaches (1:5, sample to water) prepared from Core 55 composite material and on "water blank" samples (i.e., T-102 Field Blank, HLRF Hot Cell Blank, and HLRF DIW Blank). The pH was determined following procedure PNL-ALO-225, "Measurement of pH in Aqueous Solution" and the OH^- was analyzed following procedure PNL-ALO-228, "Determination of Hydroxyl (OH^-) and Alkalinity of Solutions, Leachates, and Supernates." The water leaches were conducted in the SAL and aliquots then transferred to the Inorganic Analysis Group for subsequent ISE ammonia analysis. Other than the analysis of duplicates and a blank (for the core composite), no other sample QC was performed. Based solely on the pH values, OH^- is not expected to be present in titratable concentrations in either the core composite leachates or the water blanks. As expected, no OH^- was found in any of the samples analyzed. For the core composite it is estimated that the MDL is about 90 $\mu g/g$.

Cyanide Results: Table 2-5a through 2-5d

The Total CN analyses were performed "directly" on the Core 55 composite material as well as on "water blanks" (i.e., T-102 Field Blank, HLRF Hot Cell Blank, and HLRF DIW Blank). The core composite samples were distilled following procedure PNL-ALO-285, "Total Cyanide by Remote Microdistillation and Argentometric Titration"; however, since high CN was not expected, the pretreatment steps (i.e., predissolution in EDTA and ethylenediamine) were omitted and the distillate was measured colorimetrically using a Lachat Autoanalyzer following the manufacturer's CN procedure. The distillations of the core composite samples were performed in the SAL and sample aliquots, along with the water blanks (stabilized with NaOH), then transferred to the Inorganic Analysis Group for subsequent CN colorimetric determination. The water blank samples were analyzed both with and without distillation; and after distillation with all analytical operations were performed by the Inorganic Analysis Group. Two CRA MDL level standards, 0.010 μg/mL and 0.025 μg/mL, were analyzed and recovered at 92% and 87%, respectively.

Core 55 Composite (93-08755-G) -- Table 2-5a: The RPD for the core composites is acceptable at 13%, indicating reasonable sample homogeneity. Also, both the spiked sample and the spiked blank recovered within acceptance limits. The Total CN MDL for solids has been established at 0.9 μg/g.

T-102 Field Blank (93-05874-P), HLRF Hot Cell Blank (93-09774-P), and HLRF DIW "Blank" (93-09804-P) -- Tables 2-5b through 2-5d: The water blank samples were originally analyzed without distillation, due to a misunderstanding during the transfer of the samples from the SAL to the Inorganic Analysis Group. Additional samples were obtained from the IC aliquots, distilled, and analyzed for CN. There was insufficient sample to perform both duplicate analyses and sample spike and spiking was chosen over duplicate analysis. However, all CN results, both distilled and undistilled, produced the same results. The spike recovery for the HLRF Hot Cell Blank was 124% which is slightly outside the acceptance limit. The uncertainty of the Hot Cell Blank result (which is less than the MDL) and the slightly low recoveries demonstrated by the CRA MDL standard, may contribute to the failure. The Total CN MDL for solutions has been established at 17 μg/mL.

Ammonia ISE Results: Table 2-5a through 2-5d

The Ammonia analyses were performed on water leaches prepared from Core 55 composite material and on "water blank" samples (i.e., T-102 Field blank, HLRF Hot Cell Blank, and HLRF DIW Blank). The core composite samples were leached following procedure PNL-ALO-103, "Water Leach of Sludges, Soils, and Other Solid Samples," and then analyzed following procedure PNL-ALO-226, "Ammonia (Nitrogen) in Aqueous Samples." It should be noted that no distillation procedure is performed on the samples and the Ion Specific Electrode (ISE) analysis is performed directly on the leachates. Also, ammonia is reported as μg/g nitrogen (i.e., NH_3-N), not μg/g ammonia.

The water leaches were conducted in the SAL, stabilized with H_2SO_4, and sample aliquots then transferred to the Inorganic Analysis Group for subsequent ISE ammonia analysis. Other than the analysis of duplicates and a blank (for the core composite), no other sample QC was performed. Although a matrix spike (to be added during leaching) is required, no matrix spike was processed; see Deficiency Report DR-93-033. However, post spikes were performed on each sample to evaluate potential matrix effects.

No ammonia was detected in the core composite samples or in any of the water blanks, except for 93-08755-C1 which measured ammonia above the IDL but lower than the MDL. Since no results were above the MDL, no RPDs have been calculated. For the water blanks, the IDL and MDL are 0.05 μg/mL and 0.5 μg/mL, respectively. Assuming a 100x leaching ratio, the IDL and MDL for the composite samples are 5 μg/g and 50 μg/g, respectively. The analytical post spike for all composite samples and water blanks recovered within the range of 90-100%.

/A-58

TOC/TIC/TC Results: Table 2-5a through 2-5d

The TOC/TIC/TC analyses were performed on water leaches prepared from Core 55 composite material as well as "directly" on the composite material. Also, included are "direct" TOC/TIC/TC results from the T-102 Field Blank, HLRF Hot Cell Blank, and HLRF DIW Blank. The core composite samples were leached following procedure PNL-ALO-103, "Water Leach of Sludges, Soils, and Other Solid Samples," and then analyzed following procedure PNL-ALO-382, "Solution Analysis: Carbon." Direct TOC/TIC/TC analyses on core composites and the Field, Hot Cell, and DIW blanks were performed following procedure PNL-ALO-381; "Determination of TC, TOC, and TIC in Radioactive Liquids, Soils, and Sludges by Hot Persulfate Method."

The water leaches were performed in the SAL and sample aliquots then transferred to the Inorganic Analysis Group for subsequent TOC/TIC/TC solution analysis. Other than the analysis of duplicates and a blank, no other sample QC was performed. Although a matrix spike (to be added during leaching) is required, no matrix spike was processed (see Deficiency Report DR-93-033). However, post spiking was performed to evaluate matrix interferences. The hot persulfate TOC/TIC/TC analyses were performed entirely in the SAL and include sample matrix spikes as well as the sample, duplicate, and blank.

"Water Leach" Analysis:

The RPD for the TOC, TIC, and TC for the core composites is quite good, indicating reasonable sample homogeneity. No sample spiking was performed at the time of leaching; however, post-leach analytical spike recoveries for organic carbon (potassium acid phthalate analyzed as TC) and inorganic carbon (sodium carbonate analyzed as TIC) ranged from 102% to 111%. An MDL of 50 μg/g has been established for TC and TIC; the MDL is based on the standard deviation of the system blanks and assumes a 100x leaching ratio. It should be noted that the core composite TOC results for the water leach analysis are slightly higher than those obtained from the direct hot persulfate oxidation analysis; this condition is atypical and can not be explained.

"Direct" Analysis:

The core composite RPD for TC, TOC and TIC is marginal, indicating some level of sample heterogeneity. Recovery of sample spikes for inorganic carbon (calcium carbonate analyzed as TIC) and organic carbon (glucose analyzed as TOC) were very good for a repeated spike analysis; being 117% and 106%, respectively. In contrast to other reported inorganic results, the "direct" hot persulfate oxidation TOC/TIC procedure requires that the reported results be corrected for the "system" blank levels as well as the "system" check standard recoveries. Therefore, all samples have been corrected for both the system blank and system check standard recoveries (which ranged from 92% to 98% for TIC and 91% to 95% for TOC). The carbon results for the direct analysis appear to be about 25% lower than those from the water leach. Since the direct analysis is considered to provide a "total" analysis for carbon, the higher results from the water leaches can not be explained.

The Field Blank, Hot Cell Blank and the DIW Blank TOC/TIC/TC analyses were all very low; typically measuring carbon at 1.5 to 2 times the "system" blank levels. At this level, the carbon results for all blanks is considered equivalent and the RPDs, not being meaningful for results at these concentrations, have not been calculated. The matrix spike analysis performed on the Field Blank recovered well at 98% for TIC and 103% for TOC.

An MDL based on the standard deviation of the system blank is used for reporting purposes and is a deviation from the QAPjP (see Deficiency Report DR-93-033. For the hot persulfate method the MDL=IDL and all results less than MDL are reported as ND. The estimated MDL for TOC, assuming samples sizes of 0.2 g for solids and 5 mL for liquids, is 80 μg/g and 3.2 μg/mL, respectively. The comparable TIC MDLs are 45 μg/g and 1.8 μg/mL.

SECTION 3

RADIOCHEMISTRY

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RADIOCHEMICAL ANALYSIS

A full suite of radiochemical analyses were performed on water leach and KOH fusion samples of the Core 55 Composite prepared by the SAL. In addition, gamma energy analyses, total beta and total alpha analyses were performed on the T-102 Field Blank, HLRF Hot-Cell Blank and HLRF Deionized Water (DIW). Gamma energy analysis was performed on a fusion prep of the water leach residue. Duplicate samples and blanks were analyzed as specified in the Test Instructions. Results are based on wet weight of the sample. The factors used to correct individual sample results for analyst yield are historical averages for standards run by the responsible analyst. These are kept in the analyst standards folder. This standard is a reagent spike processed similarly to the sample after preparation by the SAL. Reagent Blank results are based on a theoretical sample of the same size taken to a volume, which is typical of the samples, and indicates contamination that may have occurred after preparation by the SAL. Method blanks are prepared in the SAL and are based on the average weight of the samples prepared at that time and therefore indicate contamination that occurred either in sample preparation and/or the analysis.

Matrix effects were evaluated by analyzing post digestion spikes. A post digestion spike consists of an aliquot of a regular sample, after preparation in the SAL, to which a known quantity of the analyte is added. Spike recovery is based on the analyzed difference between the spiked and unspiked aliquots of the sample. Spike recoveries ranged from 85% to 103% for all samples.

The total alpha, total beta, and individual radionuclides for the core composite, water leach, water leach residue and blanks are summarized in Tables 3-1 through 3-6. The data are listed by ACL number; the sample preparation method and type of sample (i.e., sample, duplicate, matrix spike, or blank) is also given.

Gamma Energy Analysis -- Table 3-1: Gamma energy analyses of duplicate samples of the core composite prepared by caustic fusion agree quite well; the RPD for ^{137}Cs activities is 5% for the Core 55 Composite and 15% for the residue from the leached solids. The RPDs for ^{154}Eu, ^{155}Eu and ^{241}Am exceed the required 20% limits

for both the water leach (93-08755-C1 and C2) and the fusion of residual solids (FRS) of the water leach samples (93-08755-CH1 and CH2). These differences may be due to the poor water leachability of Eu from these samples, as discussed later. The RPD for ^{60}Co for the FRS samples is also high; however, the blank indicated significant ^{60}Co contamination from the hot cells.

The gamma analysis of the fusion of the residual solids from the water leach indicated that most of the europium (about 92%) and americium (about 84%) as well as part of the cesium (about 14%) was not leached by the water and remained in the residue. This is consistent with the comparison of the water leach and the fusion of the Core 55 composite which also indicates that the water leach is rather ineffective at leaching these isotopes. The sum of the cesium activities in the water leach and the fusion of the residual solids is about the same as the fusion of the composite; however, the sums for europium and americium are much lower, possibly indicating sample inhomogeneities.

Uranium Analysis -- Table 3-2: Total uranium concentrations were measured in the fusion composite samples using laser fluorimetry. The RPD between the sample and duplicate is 8%, which is within the 10% measurement uncertainty.

Isotopic Analysis -- Table 3-3: Thermal ionization mass spectrometry was used to determine the presence of all isotopes of uranium. Because of the low plutonium content of these samples, plutonium isotopic composition by mass spectrometry was not possible. However, isotopic information is available from Alpha Energy Analysis of the separated plutonium.

Total Alpha, Pu, Am/Cm, and Np Analysis -- Table 3-4: Total alpha, Pu, Am/Cm, and Np analyses were performed on KOH fusions of the core composite. Total alpha was also measured on the Water Leach, Field Blank, Hot-Cell Blanks and Hot-Cell DIW. The total alpha activity was determined by drying a small aliquot on a counting plate and counting. The Pu, Am/Cm, and Np fractions were separated by ion exchange and/or solvent extraction procedures and similarly counted. The sample preparation blank results were two to four orders of magnitude lower than the samples indicating little contamination of samples. The total alpha results agree reasonably well with the sum of the individual alpha emitting nuclides with

the total alpha being slightly lower (25%) than the sum. This difference is most likely due to alpha absorption by the sample residue on the alpha counting mount, as indicated by the post digestion spike recovery of only 85%. The plutonium analyses are reported as total alpha Pu and since the Pu concentration of the samples was too low for isotopic determination by Mass Spectrometry, $^{239-240}Pu$ and ^{238}Pu from Alpha Energy Analysis of the separated Pu are also reported. Agreement of duplicates for the alpha emitters is generally within the stated statistical uncertainties. The americium alpha results also agree with the americium activities determined by gamma energy analysis. The alpha activity of the Field Blank and HLRF DIW are below the minimum detectable activity (MDA). The HLRF Hot-Cell Blanks are more than two orders of magnitude above the MDA. Post Digestion Spike recoveries ranged from 85% for the total alpha on the fused samples to 103% on the total alpha on the water leach.

Total Beta, ^{90}Sr, and ^{99}Tc Analysis -- Table 3-5: Total Beta analyses were performed on the KOH fusion, Water Leach, Field Blank, Hot-Cell Blank and Hot-Cell DIW. Only the KOH fusion was analyzed for ^{90}Sr and ^{99}Tc. Total beta values are determined by drying a small aliquot of each solution and counting in a beta proportional counter. ^{99}Tc and ^{90}Sr were also measured by beta counting after separation of each fraction by ion exchange and/or solvent extraction. The duplicate agreement is good; the largest RPD is 15% for strontium. The preparation blank activities are orders of magnitude below those of the samples. Total beta activities are in good agreement with the sum of the individual beta emitters analyzed. Total beta activities are calculated assuming beta counter efficiencies for ^{90}Sr + ^{90}Y in equilibrium. Very low activities of ^{99}Tc were detectable. A small amount of beta activity was detected in the Field Blanks and the HLRF Hot-Cell Blank activity is almost four orders of magnitude above that of the HLRF DIW.

Tritium Analysis -- Table 3-6: The analysis of the tritium blanks shows possible gross contamination of the core composite samples during preparation in the hot cell. This has been a recurring problem due to high residual tritium levels in the SAL. Duplicate analyses do not agree very well (RPD of 24%); however, the disagreement could be caused by varying contamination during sample leaching in the SAL. In addition there was some high energy beta residual in the tritium

distillate which was not removed with a second distillation. However, analysis of the beta spectra indicated no significant contribution to the tritium activity. Because of this problem, the estimated uncertainties of these results were increased by 10%.

Carbon-14 Analysis -- Table 3-6: These Analyses were performed in a single batch. The QC acceptance criteria require that two out of three blanks to be less than 40 cpm. All three blanks met this requirement. The target for system standards recovery is for two out of three to be >80% and <110%. All three met this requirement. The target for matrix spikes, as per procedure PNL-ALO-482, "Determination of Carbon-14 in Radioactive Liquids, Soils and Sludges," is >75% and <125% recovery. The measured matrix spike result of 76% just met this requirement. The RPD for the sample and and duplicate was good at 10%. The estimated method precision and accuracy is ±10% and ±15%, respectively.

Table 3-1: T-102 Core 55, Gamma Energy Analysis (GEA) Results

GEA Results

Sample ID	*	K(40) uCi/g	Co(60) uCi/g	+/- % error	Ru(103) uCi/g	Ru(106) uCi/g	Cs(134) uCi/g	Cs(137) uCi/g	+/- % error	Ce(144) uCi/g
T-102 Field Blank										
93-05874-R1	DA/smp	<2.4E-05	<2.2E-06		<6.0E-05	<1.8E-05	<1.7E-06	<5.6E-06		<1.5E-05
93-05874-R2	DA/dup	<2.6E-05	<2.2E-06		NA	<1.6E-05	<1.8E-06	5.55E-06	40	<1.4E-05
93-05874-R5	DA/pds		96%							
T-102 HLRF Hot Cell Blank										
93-09774-R1	DA/smp	<2.5E-05	<2.7E-06		<3.7E-04	<7.3E-05	<6.4E-06	2.82E-03	4	<5.6E-05
93-09774-R2	DA/dup	<2.2E-05	<1.8E-06		<3.9E-04	<7.3E-05	<6.1E-06	2.88E-03	4	<5.8E-05
RPD								2%		
T-102 HLRF DIW Blank										
93-09804-R1	DA/smp	<3.9E-05	<3.7E-06		<1.6E-04	<2.6E-05	<2.2E-06	<3.9E-06		<1.8E-05
93-09804-R2	DA/dup	<4.6E-05	<3.3E-06		<2.2E-04	<4.7E-05	<2.2E-06	<8.4E-06		<2.2E-05
Core 55 Composite										
93-08755-H1	F/smp	<1.6E-02	2.88E-02	5	<8.8E-01	<1.6E-01	<1.3E-02	3.27E+01	4	<1.3E-01
93-08755-H2	F/dup	<1.5E-02	2.68E-02	6	<8.8E-01	<1.5E-01	<1.2E-02	3.10E+01	4	<1.3E-01
RPD			7%					5%		
93-08755-H3	F/blk	<6.3E-03	<1.2E-03		<4.4E-02	<1.1E-02	<9.0E-04	1.10E-02	12	<7.7E-03
93-08755-H4	F/pds		100%							
93-08755-C1	W/smp	<2.5E-03	8.52E-04	13	<3.7E-01	<5.5E-02	<4.4E-03	2.63E+01	4	<4.1E-02
93-08755-C2	W/dup	<1.6E-03	<4.6E-04		<3.8E-01	<5.5E-02	<4.4E-03	2.66E+01	3	<4.2E-02
RPD								1%		
93-08755-C3	W/blk	<2.7E-03	<2.2E-04		<9.4E-03	<1.7E-03	<1.3E-04	7.53E-04	31	<1.4E-03
93-08755-C4	W/pds		99%							
93-08755-CH1	FRS/smp	<9.5E-03	J 1.62E-02	7	<4.1E-01	<7.2E-02	<6.4E-03	4.74E+00	3	<6.7E-02
93-08755-CH2	FRS/dup	<9.6E-03	3.46E-02	5	<3.8E-01	<6.7E-02	<5.7E-03	4.09E+00	4	<5.9E-02
RPD			72%					15%		
93-08755-CH3	FRS/blk	<1.3E-02	2.31E-01	3	<1.9E-01	<3.4E-02	<3.3E-03	8.71E-01	4	<2.1E-02

DA = direct acidified, F = fusion, W = water leach, FRS = fusion of residual solids from water leach
smp = sample, dup = duplicate, blk = methods blank, pds = post digestion spike, NA = data not available

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Def 10-26-93

Table 3-1: T-102 Core 55, Gamma Energy Analysis (GEA) Results Cont'd

GEA Results

Sample ID	*	Eu(152) uCi/g	+/- % error	Eu(154) uCi/g	+/- % error	Eu(155) uCi/g	+/- % error	Th(228) uCi/g	Am(241) uCi/g	+/- % error
T-102 Field Blank										
93-05874-R1	DA/smp	<3.7E-06		<5.1E-06		<4.1E-06		<3.1E-06	<2.0E-06	
93-05874-R2	DA/dup	<3.2E-06		<3.0E-06		<4.1E-06		NA	<1.9E-06	
93-05874-R5	DA/pds									
T-102 HLRF Hot Cell Blank										
93-09774-R1	DA/smp	6.71E-05	6	1.18E-04	4	4.66E-05	13	<1.3E-05	<1.3E-05	
93-09774-R2	DA/dup	7.32E-05	7	1.20E-04	5	4.03E-05	14	<1.4E-05	<1.3E-05	
RPD		9%		2%		15%				
T-102 HLRF DIW										
93-09804-R1	DA/smp	<3.9E-06		<7.0E-06		<4.9E-06		<4.6E-06	<2.9E-06	
93-09804-R2	DA/dup	<1.1E-05		<8.7E-06		<5.4E-06		<5.5E-06	<3.2E-06	
Core 55 Composite										
93-08755-H1	F/smp	<9.0E-03		4.97E-01	2	5.57E-01	4	<3.1E-02	2.11E-01	7
93-08755-H2	F/dup	<8.0E-03		4.82E-01	2	5.28E-01	6	<3.1E-02	2.46E-01	7
RPD				3%		5%			5%	
93-08755-H3	F/blk	<3.3E-03		<2.7E-03		<2.2E-03		<1.7E-03	<1.1E-03	
93-08755-H4	F/pds									
93-08755-C1	W/smp	<4.4E-04		J 1.86E-02	4	J 1.81E-02	15	<1.1E-02	J 6.57E-03	46
93-08755-C2	W/dup	<1.2E-03		1.20E-02	6	1.30E-02	18	<1.1E-02	1.44E-02	21
RPD				43%		27%			75%	
93-08755-C3	W/blk	<7.8E-04		<3.4E-04		<4.1E-04		<3.4E-04	<2.0E-04	
93-08755-C4	W/pds									
93-08755-CH1	FRS/smp	<5.6E-03		J 1.90E-01	2	J 2.15E-01	4	<1.4E-02	8.29E-02	10
93-08755-CH2	FRS/dup	<6.6E-03		1.52E-01	3	1.65E-01	5	<1.2E-02	7.31E-02	10
RPD				22%		26%			13%	
93-08755-CH3	FRS/blk	<4.3E-03		<5.6E-03		<5.8E-03		<5.7E-03	3.03E-03	43

DA = direct acidified, F = fusion, W = water leach, FRS = fusion of residual solids from water leach
smp = sample, dup = duplicate, blk = methods blank, pds = post digestion spike, NA = data not available

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Def 10-26-93

Table 3-3: T-102 Core 55, Uranium Isotopic Analysis Results

Uranium Isotopic Analysis Results

Sample ID	*	U-234	U-235	U-236	U-238
Core 55 Composite					
93-08755-H1	F/smp	0.006	0.689	0.013	99.292
93-08755-H2	F/dup	0.005	0.706	0.013	99.276
93-08755-H3	F/blk				
93-08755-H6	F/pds				

Sample ID	*	Pu-238	Pu-239	Pu-240	Pu-241	Pu-242
Core 55 Composite						
93-08755-H1	F/smp	Pu concentration too low for Mass Spec analysis.				
93-08755-H2	F/dup					
93-08755-H3	F/blk					
93-08755-H6	F/pds					

F = fusion, smp = sample, dup = duplicate, blk = methods blank, pds = post digestion spike

Alpha Analysis Results

Sample ID	*	Tot Alpha uCi/g	+/- % error	Alpha Pu uCi/g	+/- % error	Pu-239/240 uCi/g	+/- % error	Pu-238 uCi/g	+/- % error	Am-241 uCi/g	+/- % error	Np-237 uCi/g	+/- % error
T-102 Field Blank													
93-05874-R1	DA/smp	J <9E-07											
93-05874-R2	DA/dup	<9E-07											
93-05874-R5	DA/pds	98%											
HLRF Hot Cell Blank													
93-09774-R1	DA/smp	J 1.08E-05	11										
93-09774-R2	DA/dup	1.30E-05	10										
RPD		18%											
93-09774-R5	DA/pds	9.90E+01											
HLRF DIW													
93-09804-R1	DA/smp	J <9E-07											
93-09804-R2	DA/dup	<7E-07											
Core 55 Composite													
93-08755-C1	W/smp	J 5.82E-03	9										
93-08755-C2	W/dup	6.60E-03	8										
RPD		13%											
93-08755-C3	W/blk	<7E-05											
93-08755-C6	W/pds	97%											
93-08755-H1	F/smp	J 2.27E-01	5	J 6.59E-02	15	J 5.96E-02	13	J 6.30E-03	15	J 2.44E-01	9	J 6.36E-04	16
93-08755-H2	F/dup	2.30E-01	5	5.64E-02	15	5.10E-02	14	5.38E-03	21	2.69E-01	9	4.40E-04	20
RPD		1%		16%		16%		16%		10%		36%	
93-08755-H3	F/blk	<3E-04		1.50E-04	64					<4E-04		<1E-04	
93-08755-H6	F/pds	85%		91%						104%		97%	
STANDARD										99%			
BLANK										1.60E-03	35		

DA = direct acidified, W = water leach, F = fusion
smp = sample, dup = duplicate, blk = methods blank, pds = post digestion spike

Table 3-4: T-102 Core 55, Alpha Analysis Results

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Table 3-4: T-102 Core 55, Alpha Analysis Results Cont'd

Alpha Analysis Results

Sample ID	*	Cm-243+244 uCi/g	+/- % error
Core 55 Composite			
93-08755-H1	F/smp	J 1.40E-03	33
93-08755-H2	F/dup	1.10E-03	43
RPD		24%	
93-08755-H3	F/blk		
93-08755-H6	F/pds		

F = fusion
smp = sample, dup = duplicate, blk = methods blank, pds = post digestion spike

10-26-93

Table 3-5: T-102 Core 55, Beta Analysis Results

Beta Analysis Results

Sample ID	*	Tot Beta uCi/g	+/- % error	Sr-90 uCi/g	+/- % error	Tc-99 uCi/g	+/- % error
T-102 Field Blank							
93-05874-R1	DA/smp	1.05E-05	33				
93-05874-R2	DA/dup	1.23E-05	29				
RPD		16%					
93-05874-R5	DA/pds	95%					
HLRF Hot Cell Blank							
93-09774-R1	DA/smp	4.41E-02	3				
93-09774-R2	DA/dup	4.42E-02	3				
RPD		0%					
93-09774-R5	DA/pds	91%					
HLRF DIW							
93-09804-R1	DA/smp	5.40E-06	60				
93-09804-R2	DA/dup	<5E-06					
Core 55 Composite							
93-08755-C1	W/smp	3.95E+01					
93-08755-C2	W/dup	3.53E+01					
RPD		11%					
93-08755-C3	W/blk	4.69E-02					
93-08755-C6	W/pds	95%					
93-08755-H1	F/smp	4.90E+02		2.56E+02	7	1.70E-02	13
93-08755-H2	F/dup	4.87E+02		2.20E+02	8	1.88E-02	12
RPD		1%		15%		10%	
93-08755-H3	F/blk	1.19E-02		<3E-02		<2E-03	
93-08755-H6	F/pds	92%		92%		101%	

DA = direct acidified, W = water leach, F = fusion
smp = sample, dup = duplicate, blk = methods blank, pds = post digestion spike

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Table 3-6: T-102 Core 55, Liquid Scintillation Counting Analysis Results

Liquid Scintillation Counting Analysis Results

Sample ID	*	C-14 uCi/g	+/- % error	H-3 uCi/g	+/- % error
Core 55 Composite					
93-08755-C1	W/smp	4.9E-02	15	7.94E-03	14
93-08755-C2	W/dup	4.4E-02	15	6.25E-03	15
RPD		10%		24%	
93-08755-C3	W/blk	1.5E-02	15	1.71E-03	16
93-08755-C6	W/pds			89%	
STANDARD				94%	
BLANK	REAGENT			6.60E-05	43

W = water leach
smp = sample, dup = duplicate, blk = methods blank, spk = matrix spike, pds = post digestion spike

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VALIDATION NARRATIVE

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WHC-SD-WM-DP-052-
ADDENDUM 1 REV. 0

T-102 DATA VALIDATION REPORT
CORES 55 AND 56
ANALYZED BY PACIFIC NORTHWEST LABORATORY

Westinghouse Hanford Company
P. O. Box 1970
Richland, Washington 99352

October 26, 1993

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T-102 Cores 55 and 56 Data Validation

Validation of the Tank 241-T-102, Cores 55 and 56 data package was performed to the requirements provided in Sections 2.0-2.4, of WHC-CM-5-3, Rev. 0. The overriding QA document was Appendix D of WHC-SD-PLN-047, Rev. 1. The report forms listed in the WHC-CM-5-3 manual section 2.0 and 2.4 were not used for this report. Instead, this report has been written to provide the data user a narrative that incorporates all the required aspects that would be included on the validation forms. The sample analyses were performed by the Pacific Northwest Laboratory (PNL) 325 Analytical Chemistry Laboratory (ACL) staff.

The primary objective of the data validation effort was to ensure the usability and defensibility of the data produced for the Single Shell Tank (SST) characterization project. This was accomplished through a detailed examination of the data package to recreate the analytical process and verify that proper and acceptable analytical techniques had been applied. Additionally, the data package was checked for correct submission of required deliverables, correct data transcriptions from the raw data to the data summary forms, and for proper calculation of a number of parameters. An overall assessment of the data for each Sample Data Group (SDG) is provided on the Data Assessment Summary Form as required by WHC-CM-5-3.

Data Assessment Summary Tables

Data Assessment Summary Tables are contained both at the end of this narrative, and individually in each appropriate section of analysis validation. The Summary Tables present the data qualifiers and sub-qualifiers assigned to all analytical results and include all properties and analytes reported for the segments, cores, composites and drainable liquids. The analytical results included on the Summary Tables are taken from the raw data sheets and are presented in a more condensed form than in the Sample Data Summary. The Summary Tables include specific qualification categories and present a concise package of all data validation results.

Each table identifies the type of analysis, the composite or segment analyzed, the unique sample ID, and the type of digestion, if appropriate. The analyte or property and the corresponding results are listed in the first two columns. If the sample results are below the instrument detection limit (IDL), a "less than" symbol followed by the IDL is reported. When sample results have been qualified as unreliable, an "R" is placed after the value in the results column to provide a quick reference to unreliable data.

The numbered qualifier columns at the top of the page provide a more comprehensive presentation of the assessments made to each sample result. Each numbered column corresponds to a separate qualification category; while each qualification category does not apply to all analyses, the qualification numbers always refer to a specific qualification criteria. The qualifier that appears in a given category indicates that a particular qualification criteria was not met for that analyte. For example, if a "J" qualifier is in the number "9" column for arsenic, this would indicate that arsenic was qualified as estimated due to duplicate analysis failure.

Validation of the chemical analyses data package was performed to the requirements provided in Section 2.0 of WHC-CM-5-3, Rev. 0. The qualification categories for non-radiochemical analyses are presented below:

1 Chain of Custody
2 Holding Times
3 Instrument Calibration
4 Initial and Continuing Calibration Verification
5 Analytical Blanks
6 Preparation Blanks
7 Interference Check Sample
8 Laboratory Control Sample
9 Duplicate Analysis
10 Matrix Spike or Post-Digestion Spike
11 Retention Time
12 Contract Required Detection Limit Standard
13 Serial Dilution

Validation of the alpha plutonium and isotopic uranium and plutonium analyses of the data package was performed to the requirements provided in Section 2.4 of WHC-CM-5-3, Rev. 0. The unique qualification categories for these evaluations listed below:

1 Chain of Custody
2 Initial Calibration
3 Efficiency Checks
4 Background Checks
5 Preparation Blank
6 Laboratory Control Sample
7 Duplicate Analysis
8 Matrix Spikes/Tracers/Carriers

When Quality Assurance criteria are not met in a particular category for a sample result, the appropriate data qualifier is attached. By cross-referencing the above lists, one can see which qualification criteria were lacking. The RCRA validation process data qualifiers are defined as follows:

U The material was analyzed for, but was not detected. The associated value is the MDL or SQL.

UJ The material was analyzed for, but was not detected. The MDL or SQL is an estimated quantity.

J The associated value is an estimated quantity.

R The data are unusable.

Chemical Data Validation Narrative

Inductively Coupled Argon Plasma (ICP)

Samples from each core composite and two segments were analyzed by ICP. Sample preparation consisted of either acid digestion, KOH fusion, or water leach. The metals were determined by simultaneous ICP using procedure PNL-ALO-211. All ICP analyses were corrected for interelement spectral interferences.

High Method Detection Limits (MDLs) were reported for the fusion ICP results. The high MDLs resulted from the very high sample dilution (>11000X) used by the lab. The higher detection limits increased the significance of the process blank which had a dilution factor of 1.0. Because of this effect, there are a number of false positives reported for the fused samples. These false positives included a number of RCRA TCLP metals which, if reported, will indicate a potential to exceed the TCLP threshold (assuming a 1:20 dilution).

The ICP results for the acid digests were affected to a lesser extent. The dilution factor of approximately 400X caused fewer false positives to be reported. However the MDLs for some analytes are still above the 20X RCRA limit for some metals including cadmium, lead, and selenium. In addition, arsenic was not reported for either preparation. Due to the lack of ICV/CCV, matrix spike, and spiked blank analyses with the acid digestions, the following analytes were assigned R qualifiers with respect to each aforementioned category: B, Ce, Co, Cu, Dy, Eu, Gd, La, Li, Mg, Mo, Nd, Pd, Rh, Ru, Sn, Sr, Te, Th, Ti, Tl, W, Y, and Zn. The bismuth and silicon results were deemed unusable due to inadequate ICV/CCV recoveries. The fusion and water leach results for the same analytes were qualified as unusable because of failure to run ICV/CCV standards.

Mercury by Cold Vapor Atomic Absorption Spectrometry

Mercury was determined by cold vapor atomic absorption spectrometry (CVAA) using a modification of procedure PNL-ALO-213. All Hg results were qualified as estimated for missed hold times. In addition, results were qualified as estimated for duplicate analysis failure and matrix spike recoveries outside of the required acceptance limits.

Ion Chromatography

Anions and cyanide were determined by ion chromatography (IC) on the core composite and hot cell, field, and DIW blanks by procedure PNL-ALO-212. The samples were collected on 3/25, 3/26, and 3/28 1993 and analyzed for IC anions on 8/25/93, which is 150 days over the aqueous matrix recommended maximum holding time. All IC results have been qualified as estimated for exceeding the maximum holding time. Results were also qualified as estimated for LCS and duplicate analysis failure. As noted in the laboratory narrative, there is a matrix interference that may affect the fluoride and chloride results.

Hexavalent Chromium

Hexavalent chromium was determined spectrophotometrically on water leach samples from the core composite material using procedure PNL-ALO-227. The use of this procedure is a deviation from the protocol established in the QAPjP; the rational for the deviation is documented in Deficiency Report DR-93-041. The maximum hold time of 24 hours was grossly exceeded for the hexavalent chromium analysis. All results were qualified as estimated. In addition, results were qualified as estimated for duplicate analysis failure and failure to analyze an ICV and CCV (See DR-93-041).

Ammonia

The ammonia analyses were performed on the water leaches prepared from Core 55 composite material and on the field blank, hot cell blank, and Deionized Water (DIW) blank using procedure PNL-ALO-226. A matrix spike was not processed with the sample as documented in Deficiency Report DR-93-033. Results were qualified as estimated for missed hold times and duplicate analysis failure. All other QC criteria were met.

TOC/TIC/TC

Total organic carbon (TOC), Total Inorganic Carbon (TIC), and Total Carbon (TC) were determined on the water leaches prepared from Core 55 composite material as well as directly on the composite. Additionally, TOC/TIC/TC was determined on the field blank, hot cell blank, and DIW. The core composites were leached following procedure PNL-ALO-103 and then analyzed by procedure PNL-ALO-382. Direct analyses were performed by procedure PNL-ALO-381. All TOC/TIC/TC results were qualified as estimated for missed hold times. The direct carbon analysis were qualified as estimated for ICV/CCV, LCS, duplicate analysis, and matrix spike failures.

pH/OH

The pH and OH were determined on the Core composite material as well as hot cell blank, field blank, and DIW blank. All results were qualified as estimated for missed hold times. All other QC criteria were met.

Cyanide

Cyanide was determined on the Core 55 composite material, field blank, hot cell blank, and DIW blank. Results were qualified as estimated for missed hold times. All other QC criteria were met.

Radiochemical Data Validation Narrative

Total Alpha

Total alpha activity was determined on the KOH fusion samples for both core composites. Total alpha analyses were also performed on the water leach samples from the core composites. The sum of the alpha isotopes was approximately 75% of the total alpha activity. This discrepancy, in combination with the low (85%) matrix spike recovery indicates a possible low bias due to matrix absorption due to high solids content. Efficiency and background check results and corresponding control charts were submitted with the data package. All detectors were in control during the analyses. Initial calibration documentation has not been submitted. Therefore all total alpha results were qualified as estimated. All other QC criteria were met.

Total Beta

Total Beta activity was determined on the KOH fusion and water leach core composite samples. Efficiency and background check results and corresponding control charts were submitted with the data package. All detectors were in control during the analyses. Initial calibration documentation has not been submitted. Therefore all total beta results were qualified as estimated. All other QC criteria were met.

Americium-241

Americium-241 was determined on the KOH fusion solid core composite samples by alpha proportional counting. Efficiency and background check results and corresponding control charts were submitted with the data package. All detectors were in control during the analyses. Initial calibration documentation has not been submitted. Therefore all Am-241 results were qualified as estimated.

Neptunium-237

Neptunium-237 was determined on the KOH fusion core composite samples by alpha proportional counting. Each sample was spiked with Np-237 and all recoveries were acceptable. However the spiking levels for Np-237 were very high relative to the sample activity. In the case of sample 93-8755-H1, the spike activity was 8,000 times the sample activity. Section 2.4 of WHC-CM-5-3 suggests that spiking be performed at 0.5 to 2.0 times the sample activity. Efficiency and background check results and corresponding control charts were submitted with the data package. All detectors were in control during the analyses. Initial calibration documentation has not been submitted. Therefore all Np-237 results were qualified as estimated. All other QC criteria were met.

Strontium-90

Strontium-90 was determined on the fused samples by separation followed by beta counting. Efficiency and background check results and corresponding control charts were submitted with the data package. All detectors were in control during the analyses. Initial calibration documentation has not been

submitted. Therefore all Sr-90 results were qualified as estimated. All other QC criteria were met.

Technicium-99

Technicium-99 was determined on the drainable liquid and fused solid core composite samples by beta proportional counting. All calibration information and control charts have been submitted. All QC criteria were met.

Tritium

Tritium (H-3) was determined on the water digests by liquid scintillation. Matrix spikes were run on all samples and all recoveries were acceptable. Efficiency checks, background checks, control charts, and initial calibration documentation has been provided. All QC criteria were met.

Carbon-14

Carbon-14 was also determined on the water digests by liquid scintillation counting using internal standards. All calibration information, raw data, and standard and spike recoveries were included in the data package.

Total Uranium

Uranium was determined by laser fluorescence on the fused core composite samples. An internal standard was run with each sample; therefore, no initial calibration was required. Standards used to prepare LCS and calibration standards were not traceable to NIST. No major problems were found.

Gamma Energy Analyses (GEA)

Gamma Energy Analysis was run on duplicate samples of the fused core composite samples, water leachates of the core composites, and hot cell, field blank, and DIW blank. No matrix spikes or tracers are required for GEA determinations. Some results were qualified as estimated because of duplicate analysis failures. No significant deficiencies were found with the GEA determinations.

Mass Spectrometry

Isotopic uranium and plutonium were determined by Thermal Ionization Mass Spectrometry on the fused solid core composite samples. Matrix spikes are not required since only ratios are measured. Because of the low plutonium content of these samples, plutonium isotopic analysis was not performed. All duplicate and check standard criteria were met.

Physical Parameters Data Validation Narrative

Wt% Solids

Percent solids was determined on samples from the core composites. The wt% solids was determined in duplicate for each sample. See the laboratory narrative for details of problems encountered during the Wt% solids determination.

Particle Size

Particle size was determined on the core composite material. No problems were found with the data.

Total Dissolved Solids and Weight Percent Oxides

Total dissolved solids and Wt% oxides were determined gravimetrically on the Core 55 composite material. No problems were noted.

Scanning thermogravimetry (STG)

Thermogravimetric Analysis was run in duplicate on the unhomogenized material from Core 55. The balance calibration was checked with a 100 mg standard and the temperature calibration was checked with alumel and perkalloy curie point magnetic transition standards. Minor endotherms were observed during this analysis. See the laboratory narrative for a detailed interpretation of the results. No problems were noted.

Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) was performed in duplicate on the unhomogenized material from Core 55. Calibration was checked through the use of an indium standard. No problems were noted.

Rheology

Sample rheology, which included shear stress and shear strength, was run on the Core 55 composite material. No problems were found with the data.

Density

Density was run on the Core 55 composite material. No problems were found with the density determinations.

Suggestions

ICP

The very high dilutions for the ICP determinations have caused the laboratory to report very high detection limits for the metals determinations. In the future, separate dilutions should be run to determine high or low concentration of analytes. As a minimum, the MDL for RCRA metals should not exceed 20 times the TCLP threshold.

Total Alpha

As mentioned in the laboratory narrative, the high solids content of the samples has caused a potentional low bias in the total alpha results. Development of efficiency factors based on an absorption curve will minimize the effect of high solids on total alpha results.

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DATA ASSESSMENT

SUMMARY TABLES

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OTHER ANALYSES
T-102 CORE 55 PHYSICAL PROPERTIES

PROPERTY	RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
CENTRIFUGED SOLIDS														
Volume %	96													
Weight %	97													
DENSITY g/mL														
As Received Sample	1.79													
1:1 Dilution	1.11													
1:3 Dilution	1.05													
Centrifuged Supernate	1.1													
Centrifuged Solids	1.8													
Wt% Total Solids	72.3													
Wt% Oxides	65.7													
WT% SOLIDS														
Extrusion Sample	74.2													
Rheology Sample	72.1													
SAL Sample	99.11													
STG Sample	98.0													

OTHER ANALYSES

CORE 55 COMPOSITE SAMPLE ID 93-08755-G

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
CN	3.9 µg/g		J											

INDUCTIVELY COUPLED PLASMA - KOH FUSION
CORE 55 COMPOSITE SAMPLE ID 93-08755-H

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
Ag	(919) µg/g				J		J							
Al	311,293													
As	NR													
B	(406)				R		J							
Ba	<111													
Be	<55													
Bi	<5,531													
Ca	(781)													
Cd	(258)				J		J	J						
Ce	<1,106				R									
Co	<111				R									
Cr	(806)													
Cu	55				R									
Dy	<553				R									
Eu	<2,212				R									
Fe	19,813													
Gd	<5,531				R									
K	N/A													
La	<553				R									
Li	<332				R									
Mg	<1,106				R									
Mn	1,010													
Mo	<332				R									
Na	34,238													
Nd	(1,302)				R		J							
Ni	N/A													
P	(1,621)													
Pb	(2,008)						J							
Pd	<3,319				R									
Rh	<3,319				R									
Ru	<2,212				R									
Sb	<553													
Se	<1,106													
Si	(3,534)													
Sn	<11,062				R									
Sr	<55				R									

INDUCTIVELY COUPLED PLASMA - KOH FUSION
CORE 55 COMPOSITE SAMPLE ID 93-08755-H

		1	2	3	4	5	6	7	8	9	10	11	12	13
Te	<5,531 µg/g				R									
Th	<8,850				R									
Ti	(61)				R									
Tl	<5,531				R									
U	<22,125													
V	<111													
W	<2,212				R									
Y	<111				R									
Zn	(926)				R		J							
Zr	<111							UJ						

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

INDUCTIVELY COUPLED PLASMA - KOH FUSION

CORE 55 HOMOGENIZATION CHECK SAMPLE ID 93-8755H-Top

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
Ag	(329) µg/g						J							
Al	284,032													
As	<292													
B	(348)				R		J			J				
Ba	<37													
Be	<18													
Bi	<1,827				R									
Ca	(828)						J							
Cd	(25)						J							
Ce	<365				R									
Co	<37				R									
Cr	735													
Cu	(49)				R		J							
Dy	<183				R									
Eu	<731				R									
Fe	16,878													
Gd	<1,827				R									
K	N/A													
La	<183				R									
Li	<110				R									
Mg	<365				R									
Mn	705						J			J				
Mo	<110				R									
Na	30,160													
Nd	(389)				R		J							
Ni	N/A													
P	(952)													
Pb	(593)													
Pd	<1,096				R									
Rh	<1,096				R									
Ru	<731				R									
Sb	<183													
Se	<365													
Si	(2,647)						J							
Sn	<3,653				R									
Sr	(23)				R									

INDUCTIVELY COUPLED PLASMA - KOH FUSION
CORE 55 HOMOGENIZATION CHECK SAMPLE ID 93-8755-H TOP

		1	2	3	4	5	6	7	8	9	10	11	12	13
Te	<1,827 µg/g				R									
Th	<2,923				R									
Ti	(50)				R									
Tl	<1,827				R									
U	<7,306													
V	<37													
W	<731				R									
Y	<37				R									
Zn	(518)				R		J			J				
Zr	<37							UJ						

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

INDUCTIVELY COUPLED PLASMA - KOH FUSION

CORE 55 HOMOGENIZATION CHECK SAMPLE ID 93-8755-H BOTTOM

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
Ag	(306) µg/g						J							
Al	266,060													
As	<289													
B	(216)				R		J							
Ba	<36													
Be	<18													
Bi	<1,806				R									
Ca	(733)						J							
Cd	(19)						J							
Ce	<361				R									
Co	<36				R									
Cr	786													
Cu	(36)				R		J			J				
Dy	<181				R									
Eu	<722				R									
Fe	15,221									J				
Gd	<1,806				R									
K	N/A													
La	<181				R									
Li	<108				R									
Mg	<361				R									
Mn	1,187						J			J				
Mo	<108				R									
Na	34,682									J				
Nd	(354)				R		J							
Ni	N/A													
P	(833)													
Pb	(509)													
Pd	<1,084				R									
Rh	<1,084				R									
Ru	<722				R									
Sb	<181													
Se	<361													
Si	(2,839)						J							
Sn	<3,612				R									
Sr	(25)				R									

INDUCTIVELY COUPLED PLASMA - KOH FUSION
CORE 55 HOMOGENIZATION CHECK SAMPLE ID 93-8755-H BOTTOM

		1	2	3	4	5	6	7	8	9	10	11	12	13
Te	<1,806µg/g				R									
Th	<2,890				R									
Ti	(44)				R									
Tl	<1,806				R									
U	<7,224													
V	<36													
W	<722				R									
Y	<36				R									
Zn	(332)				R		J							
Zr	<36							UJ						

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

INDUCTIVELY COUPLED PLASMA - ACID DIGESTION
CORE 55 COMPOSITE SAMPLE ID 93-08755-A

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
Ag	(15) µg/g													
Al	145,262													
As	NR													
B	201				R		J		R	J	R			
Ba	(12)													
Be	<2													
Bi	<199				R				UJ					
Ca	625						J							
Cd	(11)													
Ce	<40				R				R		R			
Co	<4				R				R		R			
Cr	737													
Cu	(14)				R		J		R		R			
Dy	<20				R				R		R			
Eu	<80				R				R		R			
Fe	19,254													
Gd	<199				R				R		R			
K	<399								UJ					
La	<20				R				R		R			
Li	<12				R				R		R			
Mg	(107)				R		J		R		R			
Mn	755													
Mo	<12				R				R		R			
Na	27,221													
Nd	263				R				R		R			
Ni	(66)													
P	552													
Pb	374													
Pd	<120				R				R		R			
Rh	<120				R				R		R			
Ru	<80				R				R		R			
Sb	(40)													
Se	(63)													
Si	853				R						J			
Sn	<399				R				R		R			
Sr	(17)				R				R		R			

INDUCTIVELY COUPLED PLASMA - ACID DIGESTION
CORE 55 COMPOSITE SAMPLE ID 93-08755-A

		1	2	3	4	5	6	7	8	9	10	11	12	13
Te	<199 µg/g				R				R		R			
Th	<319				R				R		R			
Ti	(9)				R				R		R			
Tl	<199				R				R		R			
U	<798													
V	<4													
W	<80				R				R		R			
Y	(6)				R				R		R			
Zn	98				R				R	J	R			
Zr	42							J						

INDUCTIVELY COUPLED PLASMA - WATER LEACH
CORE 55 COMPOSITE SAMPLE ID 93-08755-C

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
Ag	<2 µg/g													
Al	950										J			J
As	NR													
B	<4				R				R		R			
Ba	<2													
Be	<1													
Bi	<101				R						UJ			
Ca	(14)								J		J			
Cd	<1													
Ce	<20				R				R		R			
Co	<2				R				R		R			
Cr	767													
Cu	<1				R				R		R			
Dy	<10				R				R		R			
Eu	<40				R				R		R			
Fe	130									J	J			
Gd	<101				R				R		R			
K	<202													
La	<10				R				R		R			
Li	<6				R				R		R			
Mg	<20				R				R		R			
Mn	10										J			
Mo	(7)				R				R		R			
Na	28,121													
Nd	<10				R				R		R			
Ni	<6													
P	408													
Pb	<12													
Pd	<60				R				R		R			
Rh	<60				R				R		R			
Ru	<40				R				R		R			
Sb	<10													
Se	<20													
Si	(45)				R						J			
Sn	<202				R				R		R			
Sr	<1				R				R		R			

INDUCTIVELY COUPLED PLASMA - WATER LEACH
CORE 55 COMPOSITE SAMPLE ID 93-08755-C

		1	2	3	4	5	6	7	8	9	10	11	12	13
Te	<101µg/g				R				R		R			
Th	<161				R				R		R			
Ti	<1				R				R		R			
Tl	<101				R				R		R			
U	<403													
V	<2										UJ			
W	<40				R				R		R			
Y	<2				R				R		R			
Zn	<4				R				R		R			
Zr	<2							UJ			UJ			

INDUCTIVELY COUPLED PLASMA - ACIDIFIED
HLRF HOT CELL BLANK SAMPLE ID 93-09774-R

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
Ag	<0.010 µg'g				UJ									
Al	<0.060													
As	NR													
B	<0.020				R									
Ba	<0.010													
Be	<0.005													
Bi	<0.500													
Ca	0.513													
Cd	(0.009)				J			J						
Ce	<0.100				R									
Co	<0.010				R									
Cr	<0.020													
Cu	(0.015)				R									
Dy	<0.050				R									
Eu	<0.200				R									
Fe	(0.038)													
Gd	<0.500				R									
K	<1.000													
La	<0.050				R									
Li	<0.030				R									
Mg	<0.100				R									
Mn	(0.028)													
Mo	<0.030				R									
Na	5,212													
Nd	(0.126)				R									
Ni	(0.049)													
P	(0.139)													
Pb	<0.060													
Pd	<0.300				R									
Rh	<0.300				R									
Ru	<0.200				R									
Sb	<0.050													
Se	<0.100													
Si	(0.143)													
Sn	<1.000				R									
Sr	<0.005				R									

INDUCTIVELY COUPLED PLASMA - ACIDIFIED
HLRF HOT CELL BLANK SAMPLE ID 93-09774-R

		1	2	3	4	5	6	7	8	9	10	11	12	13
Te	<0.500 µg/g				R									
Th	<0.800				R									
Ti	<0.005				R									
Tl	<0.500				R									
U	<2.000													
V	<0.010													
W	<0.200				R									
Y	<0.010				R									
Zn	(0.060)				R									
Zr	<0.010							UJ						

INDUCTIVELY COUPLED PLASMA - ACIDIFIED
HLRF DIW SAMPLE ID 93-09804-R

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
Ag	<0.010 µg/g				UJ									
Al	<0.060													
As	NR													
B	<0.020				R									
Ba	<0.010													
Be	<0.005													
Bi	<0.500													
Ca	<0.050													
Cd	<0.005				UJ			UJ						
Ce	<0.100				R									
Co	<0.010				R									
Cr	<0.020													
Cu	(0.017)				R									
Dy	<0.050				R									
Eu	<0.200				R									
Fe	(0.013)													
Gd	<0.500				R									
K	(1.192)													
La	<0.050				R									
Li	<0.030				R									
Mg	<0.100				R									
Mn	(0.007)													
Mo	<0.030				R									
Na	<0.080													
Nd	(0.190)				R									
Ni	<0.030													
P	<0.100													
Pb	<0.060													
Pd	<0.300				R									
Rh	<0.300				R									
Ru	<0.200				R									
Sb	<0.050													
Se	<0.100													
Si	(0.108)													
Sn	<1.000				R									
Sr	<0.005				R									

INDUCTIVELY COUPLED PLASMA - ACIDIFIED
HLRF DIW SAMPLE ID 93-09804-R

		1	*2	3	4	5	6	7	8	9	10	11	12	13
Te	<0.500 µg/g				R									
Th	<0.800				R									
Ti	<0.005				R									
Tl	<0.500				R									
U	<2.000													
V	<0.010													
W	<0.200				R									
Y	<0.010				R									
Zn	(0.119)				R									
Zr	<0.010							UJ						

INDUCTIVELY COUPLED PLASMA - ACIDIFIED
FIELD BLANK SAMPLE ID 93-05874-R

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
Ag	<0.010 µg/g				UJ									
Al	<0.060													
As	NR				R									
B	0.268													
Ba	<0.010													
Be	<0.005													
Bi	<0.500													
Ca	0.510													
Cd	<0.005				UJ			UJ						
Ce	<0.100				R									
Co	<0.010				R									
Cr	<0.020													
Cu	<0.005				R									
Dy	<0.050				R									
Eu	<0.200				R									
Fe	<0.010													
Gd	<0.500				R									
K	<1.000													
La	<0.050				R									
Li	<0.030				R									
Mg	<0.100				R									
Mn	(0.012)													
Mo	<0.030				R									
Na	2.277													
Nd	(0.170)				R									
Ni	<0.030													
P	<0.100													
Pb	<0.060													
Pd	<0.300				R									
Rh	<0.300				R									
Ru	<0.200				R									
Sb	<0.050													
Se	<0.100													
Si	1.964													
Sn	<1.000				R									
Sr	<0.005				R									

INDUCTIVELY COUPLED PLASMA - ACIDIFIED
FIELD BLANK SAMPLE ID 93-05874-R

		1	2	3	4	5	6	7	8	9	10	11	12	13
Te	<0.500 µg/g				R									
Th	<0.800				R									
Ti	<0.005				R									
Tl	<0.500				R									
U	<2.000													
V	<0.010													
W	<0.200				R									
Y	<0.010				R									
Zn	(0.037) .				R									
Zr	<0.010							UJ						

OTHER ANALYSES
CORE 55 COMPOSITE SAMPLE ID 93-08755-J

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
DIRECT ANALYSIS														
TOC	520 µg/g		J		J				J	J	J			
TIC	2280		J		J				J	J	J			
TC	2800		J		J				J	J	J			

OTHER ANALYSES

FIELD BLANK SAMPLE ID 93-05874-P

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
pH	8.5		J											
OH	ND		U J				U J							
CN	ND		U J											
DIRECT ANALYSIS														
TOC	11 µg/g		J		J				J	J	J			
TIC	6		J		J				J	J	J			
TC	18		J		J				J	J	J			

OTHER ANALYSES

HLRF HOT CELL BLANK SAMPLE ID 93-09774-P

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
pH	8.4		J											
OH	ND		U J				U J							
CN	(0.011)		J											
DIRECT ANALYSIS														
TOC	3 µg/g		J		J				J	J	J			
TIC	ND		U J		U J				U J	U J	UJ			
TC	3		J		J				J	J	J			

OTHER ANALYSES
HLRF DIW SAMPLE ID 93-09804-P

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
pH	7.9		J											
OH	ND		U J				U J							
CN	ND		U J											
DIRECT ANALYSIS														
TOC	9 µg/g		J		J				J	J	J			
TIC	3		J		J				J	J	J			
TC	11		J		J				J	J	J			

ION CHROMATOGRAHPY

CORE 55 COMPOSITE SAMPLE ID 93-08755-C

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
IC														
F	220 µg/g		J						J					
Cl	300		J						J					
NO_2	8,000		J						J					
NO_3	34,000		J						J					
PO_4	1,110		J						J					
SO_4	1,620		J						J					
WATER LEACH														
TOC	650 µg/g		J			J								
TIC	3,500		J			J								
TC	4,150		J			J								
Cr(VI)	741		J		J				J					
NH_3-N	(27)		UJ							J				

OTHER ANALYSES

HLRF HOT CELL BLANK SAMPLE ID 93-09774-R

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
NH_3-N	ND µg/g		U J							U J				

OTHER ANALYSES
HLRF DIW SAMPLE ID 93-09804-R

ANALYTE	SAMPLE RESULTS	QUALIFIERS											
		1	2	3	4	5	6	7	8	9	10	11	12	13
NH_3-N	ND µg/g		U J							U J				

OTHER ANALYSES
FIELD BLANK SAMPLE ID 93-05874-R

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
NH_3-N	ND µg/g		U J							U J				

OTHER ANALYSES
CORE 55 COMPOSITE SAMPLE ID 93-08755-M

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
pH	9.8		J											
OH	ND		U J				U J							

ION CHROMATOGRAHPY

FIELD BLANK SAMPLE ID 93-05874-Q

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
IC														
F	<0.25 µg/g		UJ						UJ					
Cl	0.3		J						J					
NO_2	<0.5		UJ						UJ					
NO_3	0.7		J						J	J				
PO_4	<0.5		UJ						UJ					
SO_4	0.7		J						J					

ION CHROMATOGRAHPY

HLRF HOT CELL SAMPLE ID 93-09774-Q

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
IC														
F	<0.25 µg/g		UJ						UJ					
Cl	1.0		J						J					
NO_2	<0.5		UJ						UJ					
NO_3	1.1		J						J					
PO_4	<0.5		UJ						UJ					
SO_4	1.9		J						J					

ION CHROMATOGRAHPY

HLRF DIW SAMPLE ID 93-09804-Q

ANALYTE	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
IC														
F	0.5 µg/g		J						J					
Cl	<0.251.0		UJ						UJ					
NO_2	<0.5		UJ						UJ					
NO_3	<0.5		UJ						UJ					
PO_4	<0.5		UJ						UJ					
SO_4	0.9		J						J					

COLD VAPOR ATOMIC ABSORPTION
ANALYTE - MERCURY

SAMPLE ID	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
93-08755-D	CORE 55 COMPOSITE													
Hg	7.7 µg/g		J							J	J			

RADIOCHEMICAL ANALYSIS

CORE 55 COMPOSITE SAMPLE ID 93-08755-C

ANALYTE	SAMPLE RESULTS	QUALIFIERS														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
TOTAL ALPHA	0.00582 µCi/g		J													
TOTAL BETA	39.5		J													
GEA																
K-40	<0.0025 µCi/g															
Co-60	0.000852															
Ru-103	<0.37															
Ru-106	<0.055															
CS-134	<0.0044															
Cs-137	26.3															
Ce-144	<0.041															
Eu-152	<0.00044															
Eu-154	0.0186							J								
Eu-155	0.0181							J								
Th-228	<0.011															
Am-241	0.00657							J								
LIQUID SCINT	uCi/g															
C-14																
H-3																

RADIOCHEMICAL ANALYSIS

CORE 55 COMPOSITE SAMPLE ID 93-08755-H

ANALYTE	SAMPLE RESULTS	QUALIFIERS														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
TOTAL ALPHA	0.227 µCi/g		J													
TOTAL BETA	490		J													
GEA																
K-40	<0.016 µCi/g															
Co-60	0.0288															
Ru-103	<0.88															
Ru-106	<0.16															
CS-134	<0.013															
Cs-137	32.7															
Ce-144	<0.13															
Eu-152	<0.0090															
Eu-154	0.497															
Eu-155	0.557															
Th-228	<0.031															
Am-241	0.233															
AEA																
ALPHA Pu	0.0659 µCi/g		J													
Pu-239/240	0.0596		J													
Pu-238	0.00630		J													
Am-241	0.244		J													
Np-237	0.000636		J													
Cm-243+244	0.00140		J													
BETA																
Sr-90	256		J													
Tc-99	0.0170															

RADIOCHEMICAL ANALYSIS

CORE 55 COMPOSITE SAMPLE ID 93-08755-CH

ANALYTE	SAMPLE RESULTS	QUALIFIERS														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
GEA	uCi/g															
K-40	<0.0095															
Co-60	0.0162							J								
Ru-103	<0.41															
Ru-106	<0.072															
CS-134	<0.0064															
Cs-137	4.74															
Ce-144	<0.067															
Eu-152	<0.0056															
Eu-154	0.190							J								
Eu-155	0.215							J								
Th-228	<0.014															
Am-241	0.0829															

RADIOCHEMICAL ANALYSIS
HLRF HOT CELL BLANK SAMPLE ID 93-09774-R

ANALYTE	SAMPLE RESULTS	QUALIFIERS														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
TOTAL ALPHA	1.08E-05 µCi/g		J													
TOTAL BETA	0.0441		J													
GEA																
K-40	<2.5E-05 µCi/g															
Co-60	<2.7E-06															
Ru-103	<3.7E-04															
Ru-106	<7.3E-05															
CS-134	<6.4E-06															
Cs-137	0.00282															
Ce-144	<5.6E-05															
Eu-152	6.71E-05															
Eu-154	1.18E-04															
Eu-155	4.66E-05															
Th-228	<1.3E-05															
Am-241	<1.3E-05															

RADIOCHEMICAL ANALYSIS

HLRF DIW BLANK SAMPLE ID 93-09804-R

ANALYTE	SAMPLE RESULTS	QUALIFIERS														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
TOTAL ALPHA	<9.0E-07 Ci/g		J													
TOTAL BETA	5.40E-06		J													
GEA																
K-40	<3.90E-05 µCi/g															
Co-60	<3.7E-06															
Ru-103	<1.6E-04															
Ru-106	<2.6E-05															
CS-134	<2.2E-06															
Cs-137	<3.9E-06															
Ce-144	<1.8E-05															
Eu-152	<8.9E-06															
Eu-154	<7.0E-06															
Eu-155	<4.9E-06															
Th-228	<4.6E-06															
Am-241	<2.9E-06															

RADIOCHEMICAL ANALYSIS
FIELD BLANK SAMPLE ID 93-05874-R

ANALYTE	SAMPLE RESULTS	QUALIFIERS														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
TOTAL ALPHA	µCi/g <9.0E-7		J													
TOTAL BETA	1.05E-5		J													
GEA	uCi/G															
K-40	<2.4E-5															
Co-60	<2.2E-6															
Ru-103	<6.0E-5															
Ru-106	<1.8E-5															
CS-134	<1.7E-6															
Cs-137	<5.6E-6															
Ce-144	<1.5E-5															
Eu-152	<3.7E-6															
Eu-154	<5.1E-6															
Eu-155	<4.1E-6															
Th-228	<3.1E-6															
Am-241	<2.0E-6															

URANIUM ANALYSIS RESULTS
CORE 55 COMPOSITE SAMPLE ID 93-08755-H1

SAMPLE ID	SAMPLE RESULTS	QUALIFIERS												
		1	2	3	4	5	6	7	8	9	10	11	12	13
93-08755-H														
Uranium	734 μg/g													

URANIUM ISOTOPIC ANALYSIS RESULTS
CORE 55 COMPOSITE SAMPLE ID 93-08755-H1

SAMPLE ID	SAMPLE RESULTS	QUALIFIERS							
		1	2	3	4	5	6	7	8
93-08755-H									
U-234	0.006								
U-235	0.689								
U-236	0.013								
U-238	99.292								

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DATA ASSESSMENT FORMS

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PHYSICAL PROPERTIES DATA ASSESSMENT

DATE: 10-18-93

REVIEWED BY: D.E. STROUP Def 10-26-93

LABORATORY: PNL

CASE #: SST 241-T-102 CORES 55 & 56

SDG #: 93-08755-PNL-102

SAMPLE/MATRIX:93-08755/SOIL
93-07987-M1/SOIL
93-07988-M1/SOIL
93-08755-K1/SOIL
93-08755-M1/SOIL
93-05874-P1/FB
93-09774-P1/HCB
93-09804-P1/DIW
93-08755-C1/SOIL

DATA ASSESSMENT SUMMARY

	Blk Den	Part Size	% Sol/Ox	pH	Rheo	TDS	DSC TGA
1. Chain of Custody	O	O	O	O	O	O	O
2. Holding Times	NA	NA	O	X	NA	NA	NA
3. Instrument Calibration	O	O	O	O	O	O	O
5. Blank Analysis	NA	NA	NA	O	NA	NA	O
8. LCS	NA	O	O	O	O	NA	O
9. Duplicate Analysis	O	O	O	O	O	O	O

O = data had no problems
X = data qualified due to minor problems
M = data qualified due to major problems, some data may be unusable

OVERALL ASSESSMENT: The samples were collected 3-25,26,& 28-93 and the pH analysis was done 8-31-93, which is over 150 days from the time of collection. It is recommended that the pH of aqueous matrices be taken immediately after collection. Although the holding time requirement for soil or sludge samples has not been established, the guidelines for aqueous matrices should be followed. The sample pH results may be the values at the time of analysis but not the true pH value of the tank contents due to the execessive holding time. The other analyses noted above are acceptable.

NOTES: The pH of the Hot Cell, Field, and DIW blanks was taken.

CYANIDE DATA ASSESSMENT

DATE 10-18-93

REVIEWED BY A. T. DiCenso ATD 10-26-93

LABORATORY PNL

CASE # SST T102-C55

SDG # 93-08755-PNL-102

SAMPLES/MATRIX
93-08755 C55
93-05874 FB
93-09774 HCB
93-09804 DIW

DATA ASSESSMENT SUMMARY

		CN			
1.	Chain of Custody	O			
2.	Holding Times	X			
3.	Instrument Calibration	O			
4.	ICV/CCV	O			
5.	Analytical Blank	N/A			
6.	Preparation Blank	O			
7.	ICS	N/A			
8.	LCS	O			
9.	Duplicate Analysis	O			
10.	Matrix Spike	O			
12*.	CRDL	N/A			
13.	Serial Dilution	N/A			

* Number 11 was intentionally omitted.
O = data had no problems
X = minor problems, data may be qualified
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The samples were collected in March of 1993, prepared by distillation, and analyzed for CN colorimetrically. Since the evaluations were not completed until August of 1993, the maximum allowable holding time specification of fourteen days was exceeded. The results were qualified accordingly. The field, hot cell, and deionized water (DIW) blanks were not distilled before their initial analyses; however, a re-analysis which followed distillation yielded comparable results. For this reason, none of the blanks were qualified with respect to the duplicate category even though a duplicate

was not run in conjunction with the distilled field, hot cell, and DIW blanks. The relative percent difference stemming from the duplicate evaluation of 93-08755 was 13%. The calibration checks, matrix spikes, and laboratory control samples (spiked blanks) associated with the analyses exhibited satisfactory recoveries.

The hot cell blank yielded a value which was greater than the IDL but less than the MDL; however, the cyanide contamination had little impact on the result of 93-08755 since the amount of cyanide in the hot cell blank was less than 1% of that found in the sample.

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

RCRA INORGANIC DATA ASSESSMENT

DATE 10/25/93 SAMPLES/MATRIX 93-08755-H1 CORE 55 COMPOSITE

REVIEWED BY M.I. Weyns-Rollosson dw 10/25/93

LABORATORY 325 PNL

CASE # 241-T-102

SDG # 93-08755-PNL-102

DATA ASSESSMENT SUMMARY

		ICP
1.	Chain of Custody	O
2.	Holding Times	O
3.	Instrument Calibration	O
4.	ICV/CCV	M
5.	Analytical Blank	O
6.	Preparation Blank	X
7.	ICS	X
8.	LCS	N/A
9.	Duplicate Analysis	O
10.	Matrix Spike	O
12.*	CRDL	N/A
13.	Serial Dilution	O

* Number 11 was intentionally omitted.

O = data had no problems
X = minor problems, data may be qualified
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The sample from Core 55 was collected on 3-25-93 and sent to the PNL 325 Shielded Analytical Laboratory (SAL) for analysis. The sample was received by SAL on 5-4-93. Analysis for ICP metals was performed on 8-23-93, within the required holding time limit of 180 days. Silver and cadmium were qualified as estimated for ICV/CCV results that exceeded control limits. The following analytes were assigned an "R" qualifier for lack of ICV/CCV check standards: B, Ce, Co, Cu, Dy, Eu, Gd, La, Li, Mg, Mo, Nd, Pd, Rh, Ru, Sn, Sr, Te, Th, Ti, Tl, W, Y, Zn. Contaminants were detected in the

ICP DATA ASSESSMENT SUMMARY CONT.

preparation blanks for Ag, B, Cd, Nd, Pb, and Zn; since the sample results were not 5 times greater than the blank results, these analytes were qualified as estimated. Cadmium and zirconium results were qualified as estimated for the interference check standard exceeding the control limit.

NOTES: The ICP analysis of sample 93-08755-H1 was performed on fusions prepared from Core 55 samples and analyzed using method PNL-ALO-211.

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0
ICP DATA ASSESSMENT

DATE 10-19-93

REVIEWED BY A. T. DiCenso 10-26-93

LABORATORY 222-S

CASE # SST T102-C55

SDG # 93-08755-PNL-102

SAMPLES/MATRIX 93-08755H-1T
93-08755H-1B

DATA ASSESSMENT SUMMARY

		ICP (KOH Fusion)				
1.	Chain of Custody	O				
2.	Holding Times	O				
3.	Instrument Calibration	O				
4.	ICV/CCV	M				
5.	Analytical Blanks	O				
6.	Preparation Blanks	X				
7.	ICS	X				
8.	LCS	N/A				
9.	Duplicate Analysis	X				
10.	Matrix Spike	N/A				
12*.	CRDL	N/A				
13.	Serial Dilution	N/A				

* Number 11 was intentionally omitted.
O = data had no problems
X = minor problems, data may be qualified
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The samples were collected in March of 1993, prepared by KOH fusion, and analyzed for ICP metals. The analyses were completed on 7-23-93. According to the governing QAPjP, matrix spike and process blank spike (LCS) analyses were not required for fusion evaluations. Due to the lack of ICV/CCV data combined with the absence of spike and LCS analyses, the following analytes were assigned R qualifiers with respect to the ICV/CCV category: B, Ce, Co, Cu, Dy, Eu, Gd, La, Li, Mg, Mo, Nd, Pd, Rh, Ru, Sn, Sr, Te, Th, Ti, Tl, W, Y, and Zn. The bismuth results were deemed unusable due to low reported ICV/CCV recoveries.

ICP DATA ASSESSMENT (cont.)

With respect to negative blank results, none exhibited an absolute value exceeding the instrument detection limit (IDL); however, the following analytes were qualified on the basis of preparation blank contamination: Ag, B, Ca, Cd, Cu, Mn, Nd, Si, and Zn. Manganese in 93-08755H-1T and Fe, Mn, and Na in 93-08755H-1B were qualified due to high relative percent difference values derived from the duplicate analyses. Zinc and boron in 93-08755H-1T and copper in 93-08755-1B were qualified since the original and duplicate values were less than the MDL and did not agree within +/- 2 times the IDL. Zirconium was considered to be estimated due to an ICS failure.

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

ICP DATA ASSESSMENT

DATE 10-22-93 SAMPLES/MATRIX 93-08755-A1

REVIEWED BY A. T. DiCenso ATD 10-26-93

LABORATORY 222-S

CASE # SST T102-C55

SDG # 93-08755-PNL-102

DATA ASSESSMENT SUMMARY

		ICP (Acid Digestion)			
1.	Chain of Custody	O			
2.	Holding Times	O			
3.	Instrument Calibration	O			
4.	ICV/CCV	M			
5.	Analytical Blanks	O			
6.	Preparation Blanks	X			
7.	ICS	X			
8.	LCS	M			
9.	Duplicate Analysis	X			
10.	Matrix Spike	M			
12*.	CRDL	N/A			
13.	Serial Dilution	O			

* Number 11 was intentionally omitted.
O = data had no problems
X = minor problems, data may be qualified
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The sample was collected in March of 1993, prepared by acid digestion, and analyzed for ICP metals. The analysis was completed on 8-25-93. Due to the lack of ICV/CCV, matrix spike, and spiked blank (LCS) data, the following analytes were assigned R qualifiers with respect to each aforementioned category: B, Ce, Co, Cu, Dy, Eu, Gd, La, Li, Mg, Mo, Nd, Pd, Rh, Ru, Sn, Sr, Te, Th, Ti, Tl, W, Y, and Zn. The bismuth and silicon results were deemed unusable due to inadequate ICV/CCV recoveries.

ICP DATA ASSESSMENT (cont.)

With respect to negative blank results, none exhibited an absolute value exceeding the instrument detection limit (IDL); however, the following analytes were qualified on the basis of preparation blank contamination: B, Ca, Cu, and Mg. Boron and Zinc were qualified due to high relative percent difference values derived from the duplicate analyses. Bismuth and potassium results were considered to be estimated due to low LCS recoveries, and silicon was noted for poor matrix spike data. The reported zirconium value was qualified as a consequence of an ICS failure. No problems were encountered with the serial dilution data. Additionally, an arsenic result was not reported for sample 93-08755-A1 due to a faulty ICP channel.

ICP DATA ASSESSMENT

DATE 10-25-93 SAMPLES/MATRIX 93-08755-C1

REVIEWED BY A. T. DiCenso ATD 10-26-93

LABORATORY 222-S

CASE # SST T102-C55

SDG # 93-08755-PNL-102

DATA ASSESSMENT SUMMARY

		ICP (Water Digestion)			
1.	Chain of Custody	O			
2.	Holding Times	O			
3.	Instrument Calibration	O			
4.	ICV/CCV	M			
5.	Analytical Blanks	O			
6.	Preparation Blanks	O			
7.	ICS	X			
8.	LCS	M			
9.	Duplicate Analysis	X			
10.	Matrix Spike	M			
12*.	CRDL	N/A			
13.	Serial Dilution	X			

* Number 11 was intentionally omitted.
O = data had no problems
X = minor problems, data may be qualified
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The sample was collected in March of 1993, prepared by water digestion, and analyzed for ICP metals. The analysis was completed on 8-23-93. Due to the lack of ICV/CCV, matrix spike, and spiked blank (LCS) data, the following analytes were assigned R qualifiers with respect to each aforementioned category: B, Ce, Co, Cu, Dy, Eu, Gd, La, Li, Mg, Mo, Nd, Pd, Rh, Ru, Sn, Sr, Te, Th, Ti, Tl, W, Y, and Zn. The bismuth and silicon results were deemed unusable due to inadequate ICV/CCV recoveries.

ICP DATA ASSESSMENT (cont.)

With respect to negative blank results, none exhibited an absolute value exceeding the instrument detection limit (IDL). Iron was qualified on the basis of a high relative percent difference value derived from the duplicate analysis, and the calcium result was considered to be estimated due to a high LCS recovery. The following analytes were noted for poor matrix spike data: Al, Bi, Ca, Fe, Mn, Si, V, and Zr. Additionally, the reported zirconium value was qualified as a consequence of an ICS failure, and the serial dilution evaluation was unsatisfactory with regard to aluminum. An arsenic result was not reported for sample 93-08755-C1 due to a faulty ICP channel.

RCRA INORGANIC DATA ASSESSMENT

DATE 10/25/93

SAMPLES/MATRIX 93-09774-R1
93-09804-R1
93-05874-R1

REVIEWED BY M.I. Weyns-Rollosson dw 10/25/93

LABORATORY 325 PNL

CASE # 241-T-102

SDG # 93-08755-PNL-102

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

DATA ASSESSMENT SUMMARY

		ICP
1.	Chain of Custody	O
2.	Holding Times	O
3.	Instrument Calibration	O
4.	ICV/CCV	M
5.	Analytical Blank	O
6.	Preparation Blank	N/A
7.	ICS	X
8.	LCS	N/A
9.	Duplicate Analysis	O
10.	Matrix Spike	O
12.*	CRDL	N/A
13.	Serial Dilution	N/A

* Number 11 was intentionally omitted.

O = data had no problems
X = minor problems, data may be qualified
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The hot cell blank, DIW blank, and the field blank samples from Core 55 were collected on 3-25-93 and sent to the PNL 325 Shielded Analytical Laboratory (SAL) for analysis. The samples were received by SAL on 5-4-93. The analysis for ICP metals was performed on 9-02-93, within the required holding time limit of 180 days. Silver and cadmium were qualified as estimated for ICV/CCV results that exceeded control limits. The following analytes were assigned an "R" qualifier for lack of ICV/CCV check standards: B, Ce, Co, Cu, Dy, Eu, Gd, La, Li, Mg, Mo, Nd, Pd, Rh, Ru, Sn, Sr, Te, Th, Ti, Tl, W, Y, Zn. Cadmium and zirconium results were qualified as estimated for the interference check standard exceeding the control limit.

NOTES: The ICP analysis of sample 93-08755-H1 was performed on fusions prepared from Core 55 samples and analyzed using method PNL-ALO-211.

TIC/TOC/TC, AMMONIA, & HYDROXYL DATA ASSESSMENT

DATE: 10-19-93

SAMPLE/MATRIX: 93-08775-J1/SOIL
93-05874-P1,R1/HCB
93-09774-P1,R1/FB
93-09804-P1,R1/DIW
93-08775-C1/SOIL
93-08755-C1/SOIL
93-08775-M1

REVIEWED BY: D.E. STROUP DES 10-26-93

LABORATORY: PNL

CASE #: SST 241-T-102 CORES 55 & 56

SDG #: 93-08755-PNL-102

ASSESSMENT SUMMARY

	Direct TIC/TOC/TC	WtrLch TIC/TOC/TC	NH_3	OH
1. Chain of Custody	O	O	O	O
2. Holding Times	X	X	X	X
3. Instrument Calibration	O	O	O	O
4. ICV/CCV	X	O	NA	NA
5. Analytical Blank	O	X	NA	NA
6. Preparation Blank	NA	NA	O	X
8. LCS	X	O	O	O
9. Duplicate Analysis	X	O	X	O
10. Matrix Spike	X	O	O	NA

O = data had no problems
X = data qualified due to minor problems
M = data qualified due to major problems, some data may be unusable

OVERALL ASSESSMENT: The samples were collected 3-25,26,& 28-93 and analyzed as follows:

Direct TIC/TOC/TC	8-18,19,& 20-93	144 days from collection
Water Leach TIC/TOC/TC	8-10-93	136 days from collection
Ammonia	9-2-93	159 days from collection
Hydroxyl Ion	9-1-93	158 days from collection

The recommended holding time for TIC/TOC and ammonia aqueous matrices is 28 days from collection of the samples. Hydroxyl Ion analysis should be done immediately after sample collection as recommended for pH. Although the requirements for soil or sludge matrices have not been established, the guidelines for aqueous matrices should be followed.

Direct TIC/TOC/TC: The analysis of the sample, duplicate, and matrix spike was done over two days (8-19,20-93) as an extended batch. The original TOC portion of the matrix spike had a recovery of 60% and the third system standard for TIC and TOC had recoveries of 61% and 62%. A fourth system standard was successfully done on the first day, and a blank, system standard, and matrix spike were successfully completed on the second day. The original TIC spike, which is run before the TOC spike, is 86%. TIC/TOC system standards, other than the previously noted third standard, produced recoveries from 92 to 98 percent for TIC and 91 to 95 percent for TOC (limits 90-110%). Other than the original TOC matrix spike of 60%, the other matrix spikes are within limits, with TIC spikes from 86 to 117 percent and TOC spikes from 103 to 106 percent (limits 75-125%). The TIC relative percent difference (RPD) is within the 20% limit, but the TOC RPD is high at 28%. The TOC results may be suspect due to the original spike recovery, excessive holding time, and high RPD. The TIC results may be suspect due to excessive holding time.

Water Leach TIC/TOC/TC: The RPDs and matrix spikes are within the recommended quality control limits noted above. Method blank results are 80 ug/g for TC and TOC, which is above the method detection limit of 50 ug/g, indicating handling contamination. These results may be suspect due to the excessive holding time.

Ammonia: The spike recoveries are within the recommended limits noted above. A difference between the sample and duplicate analysis is noted as the duplicate is non detect and a value of 27.2 ug/g is reported for the sample. There was not enough sample for a re-analysis. The results for the duplicates, field blank, hot cell blank, DIW blank are non detects and no RPDs are reported. This data may be suspect due to the excessive holding time and duplicate analysis.

Hydroxyl Ion: There was not enough sample to do the 1:1 extraction as specified and 1:5 extraction was used for the titration. No titration could be done on the field, hot cell, or DIW blanks due to the lack of a titratable constituent. The entire 5 mL portions of the 09774-P1 & P2 were combined and 2 mL of 08755-M3 were titrated with no results. No hydroxyl ion was detectable in the sample or duplicate. These results may be suspect due to the excessive holding time.

NOTES: Hot cell, field, and DIW blank analysis was done for Direct TIC/TOC/TC, Ammonia, and Hydroxyl Ion analyses.

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

IC ANIONS DATA ASSESSMENT

DATE: 10-19-93

SAMPLE/MATRIX: 93-08755-C1/SOIL
93-05874-Q1/SOIL
93-09775-Q1/SOIL
93-09804-Q1/SOIL

REVIEWED BY: D.E. STROUP DES 10-26-93

LABORATORY: PNL

CASE #: SST 241-T-102 CORES 55 & 56

SDG #: 93-08755-PNL-102

DATA ASSESSMENT SUMMARY

	IC ANIONS
1. Chain of Custody	O
2. Holding Time	X
3. Instrument Calibration	O
4. ICV/CCV	O
5. Analytical Blanks	O
8. LCS	X
9. Duplicate Analysis	X
10. Matrix Spike	O
11. Retention Time	O

O = data had no problems
X = data qualified due to minor problems
M = data qualified due to major problems, some data may be unusable

OVERALL ASSESSMENT: The recommended holding time for aqueous matrix IC Anions analysis is as follows:

Sulfate, Chloride, Fluoride	28 days
Nitrate, Nitrite, Phosphate	48 hours

The samples were collected 3-25,26,& 28-93 and IC Anions analysis was done 8-25-93, which is 150 days over the aqueous matrix recommendation. Although the requirements for soil or sludge matrices have not been established, the guidelines for aqueous matrices should be followed. The recoveries for the control standard sample are above the recommended 90-110% (see the IC Anions laboratory narrative included in this validation report for further comments). Very thorough calibration and Initial Calibration/Continuing Calibration Verifications were completed for

the analysis. Matrix spike recoveries are within the recommended 75-125%. The Relative Percent Difference value for the Field Blank sample is higher than the ±20% criteria and the Field Blank duplicate sample has a high nitrate value that the laboratory feels is due to contamination. Also check the laboratory narrative for matrix interferences on the fluoride peak and a matrix effect noted on the chloride peak that could bias the reported values.

NOTES: Hot Cell, Field, and DIW blanks were included in the analysis. This data may be suspect due to the high LCS recoveries and the excessive holding time.

MERCURY & CHROMIUM VI DATA ASSESSMENT

DATE: 10-19-93 SAMPLE/MATRIX: 93-08755-D1/SOIL
93-08755-C1/SOIL

REVIEWED BY: D.E. STROUP DES 10-26-93

LABORATORY: PNL

CASE #: SST 241-T-102 CORES 55 & 56

SDG #: 93-08755-PNL-102

ASSESSMENT SUMMARY

	Hg	CrVI
1. Chain of Custody	O	O
2. Holding Times	X	X
3. Instrument Calibration	O	O
4. ICV/CCV	NA	X
6. Preparation Blank	O	O
8. LCS	O	X
9. Duplicate Analysis	X	O
10. Matrix Spike	X	O

O = data had no problems
X = data qualified due to minor problems
M = data qualified due to major problems, some data may be unusable

OVERALL ASSESSMENT: The recommended holding times for Mercury and Chromium VI aqueous matrices are 28 days and 24 hours respectively. The samples were collected 3-25,26,& 28-93. Mercury analysis was done 8-25-93 (150 days from time of collection) and Chromium VI analysis was done 9-8-93 (164 days from time of collection). Although the requirements for soil or sludge matrices have not been established, the guidelines for aqueous matrices should be followed. No Initial Calibration and Continuing Calibration Verifications were completed for the Chromium VI analysis. A handwritten note at the bottom of the laboratory narrative refers to DR-93-041 for an explanation of the lack of this quality control check. No prespiked Chromium VI sample was analyzed, but one of the duplicate samples was post spiked with a recovery of 106%. The Mercury Relative Percent Difference for the sample and duplicate is high (46%, limit 20% - Check the Mercury laboratory narrative included in this validation report for further comments). No Mercury Matrix Spike was analyzed, but a blank spike was done with a recovery of 102%.

NOTES: The Mercury analysis was done twice, but only the results from the 8-25-93 run are reported in the laboratory data summary table. Both the Mercury and Chromium VI analyses may be suspect as the proper quality controls were not used and the holding times are excessive.

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

RADIOCHEMICAL DATA ASSESSMENT

DATE: 10-21-93

SAMPLE/MATRIX: 93-08755-C1/SOIL
93-08755-CH1/SOIL
93-08755-H1/SOIL
93-05874-R1/FB
93-09774-R1/HCB
93-09804-R1/DIW

REVIEWED BY: D.E. STROUP *DES. 10-26-93*

LABORATORY: PNL

CASE #: SST 241-T-102 CORES 55 & 56

SDG #: 93-08755-PNL-102

ASSESSMENT SUMMARY

	GEA	U	TTL A&B	Sr90	Tc99
1. Chain of Custody	O	O	O	O	O
2. Instrument Calibration	O	O	X	X	O
3. Efficiency Checks	O	O	O	O	O
4. Background Checks	O	O	O	O	0
5. Preparation Blank	O	O	O	O	O
6. LCS	O	O	O	O	O
7. Duplicate Analysis	X	O	O	O	O
8. MS/Carriers	O	O	O	O	O

O = data had no problems
X = data qualified due to minor problems
M = data qualified due to major problems, some data may be unusable

OVERALL ASSESSMENT: Initial Instrument Calibration documentation was only provided for the GEA and liquid scintillation counters. This information was not provided for the alpha and beta counters used for total alpha/beta and Sr-90 determinations. These results were qualified as estimated (J) for missing documentation. Efficiency check data and the corresponding control charts were provided for all data. Sample 08755-CH1 GEA Co60, Eu154, and Eu155; and sample 08755-C1 GEA Eu154, Eu155, and Am241 duplicate analysis Relative Percent Differences are above 20%.

RCRA HIGH LEVEL RADIOCHEMICAL DATA ASSESSMENT

DATE October 25, 1993 SAMPLES/MATRIX 93-08755-H1

REVIEWED BY D. J. Smith 10/27/93

LABORATORY PNL-325

CASE # Tank T-102

SDG # 93-08755-PNL-102

DATA ASSESSMENT SUMMARY

		Pu238	Pu239/240	Am241	Np237
1.	Chain of Custody	0	0	0	0
2.	Initial Calibration	X	X	X	X
3.	Efficiency Checks	0	0	0	0
4.	Background Checks	0	0	0	0
5.	Preparation Blank	0	0	0	0
6.	Laboratory Control Sample	0	0	0	0
7.	Duplicate Analysis	0	0	0	0
8.	Matrix Spike/Tracers/Carriers	0	0	0	0

0 = data had no problems
X = minor problems, data may be qualified
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: Initial calibration data for the alpha proportional or scintillation counters was not included in the data package. All results were qualified "estimated" (J). All other QC criteria were met.

NOTES: Matrix spike levels for Np-237 were extremely high relative to sample activities. In the case of sample 93-8755-H-1, the spike level was 8,000 times the sample activity. WHC-CM-5-3 suggests that spiking be performed at .5 to 2 times sample concentration or activity.

RCRA HIGH LEVEL RADIOCHEMICAL DATA ASSESSMENT

DATE	October 25, 1993	SAMPLES/MATRIX	93-08755-H1
REVIEWED BY	D. J. Smith		
LABORATORY	PNL-325		
CASE #	Tank T-102		
SDG #	93-08755-PNL-102		

10/27/93

DATA ASSESSMENT SUMMARY

		Cm241,243
1.	Chain of Custody	0
2.	Initial Calibration	X
3.	Efficiency Checks	0
4.	Background Checks	0
5.	Preparation Blank	0
6.	Laboratory Control Sample	0
7.	Duplicate Analysis	0
8.	Matrix Spike/Tracers/Carriers	0

0 = data had no problems
X = minor problems, data may be qualified
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: Initial calibration data for the alpha proportional or scintillation counters was not included in the data package. All results were qualified "estimated" (J). All other QC criteria were met.

NOTES:

RCRA HIGH LEVEL RADIOCHEMICAL DATA ASSESSMENT

DATE October 25, 1993

REVIEWED BY D. J. Smith 10/25/93

LABORATORY PNL-325

CASE # Tank T-102

SDG # 93-08755-PNL-102

SAMPLES/MATRIX 93-08755-C1

DATA ASSESSMENT SUMMARY

		H-3	C-14
1.	Chain of Custody	0	0
2.	Initial Calibration	0	0
3.	Efficiency Checks	0	0
4.	Background Checks	0	0
5.	Preparation Blank	0	0
6.	Laboratory Control Sample	0	0
7.	Duplicate Analysis	0	0
8.	Matrix Spike/Tracers/Carriers	0	0

0 = data had no problems
X = minor problems, data may be qualified
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: All calibration check documentation for the liquid scintillation determinations were provided. Each sample was spiked and all spike recoveries were acceptable. All other QC criteria were met.

NOTES:

RCRA HIGH LEVEL RADIOCHEMICAL DATA ASSESSMENT

DATE October 25, 1993 SAMPLES/MATRIX 93-08755-H

REVIEWED BY D. J. Smith 10/25/93

LABORATORY PNL-325

CASE # Tank T-102

SDG # 93-08755-PNL-102

DATA ASSESSMENT SUMMARY

		Iso U
1.	Chain of Custody	0
2.	Initial Calibration	0
3.	Efficiency Checks	N/A
4.	Background Checks	N/A
5.	Preparation Blank	0
6.	Laboratory Control Sample	0
7.	Duplicate Analysis	0
8.	Matrix Spike/Tracers/Carriers	N/A

0 = data had no problems
X = minor problems, data may be qualified
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: All calibration check documentation for the Thermal Ionization Mass Spec. analyses were provided. Spikes are not required since ratioes are measured. Isotopic plutonium could not be analyzed due to the low concentration in the samples. All other QC criteria were met.

NOTES:

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LETTERS OF INSTRUCTION

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Westinghouse
Hanford Company

P.O. Box 1970 Richland, WA 99352

March 29, 1993

9352556

Mr. A. G. King, Director
Analytical Chemistry Laboratory
Pacific Northwest Laboratory
Post Office Box 999
Richland, Washington 99352

Dear Mr. King:

LETTER OF INSTRUCTION FOR THE ANALYSIS OF ADDITIONAL CORE SAMPLES IN FISCAL YEAR 1993

References:
1. Draft, Technical Project Plan, "Pacific Northwest Laboratory Tank Waste Characterization Project Draft Technical Project Plan," dated March 10, 1993.
2. Letter 9302439, "Characterization Activities to Meet the M-10-07 Milestone," R. E. Gerton, RL, to President, WHC, dated March 15, 1993.
3. WHC-SOW-93-0002, Revision 0, "Sampling and Analysis of SST and DST Waste Tanks in Support of TWRS Fiscal Year 1993," dated November 1992.
4. WHC-SD-WM-PLN-047, Revision 0, "Tank Waste Remediation System Tank Waste Characterization Plan," dated December 1992.

This letter of instruction (LOI) provides changes to the core samples that are planned to be sampled during fiscal year (FY) 1993 and sent to the 222-S and 325 laboratories for analyses. The laboratory is requested to receive, extrude, and, upon approval of the technical program plan (TPP) (Reference 1), analyze the additional single-shell tank (SST) core samples described below.

Westinghouse Hanford Company (WHC) has been directed by the Department of Energy, Richland Field Office (RL) to meet the Hanford Federal Facility Agreement and Consent Order interim milestone M-10-07 by obtaining 24 core samples from 12 SSTs by September 30, 1993. While approval for extension of the 216 day requirement was not received, the RL has acknowledged that not all cores could be analyzed in this time frame (Reference 2).

Table 1 provides the core samples planned to be obtained in FY 1993 and the expected sampling completion date. Since the issuance of the statement of work (SOW) (Reference 3), five non-watch list SSTs have been added to the schedule. Due to the need to take additional SST cores and the delays in core sampling caused by the sampling stand down, sampling of double-shell tanks 241-AZ-101 and 241-AZ-102 have been deferred.

Table 1

Tank	Est. Sampling Date
241-T-107	3/10/93
241-T-105	3/23/93
241-T-102	4/2/93
241-C-106	4/19/93
241-C-104	5/3/93
241-C-105	5/12/93
241-C-111	6/7/93
241-C-108	6/28/93
241-T-101	7/21/93
241-BX-109	8/15/93
241-BX-102	9/1/93
241-AN-107	9/15/93
241-C-103	9/25/93

The cores from SSTs 241-T-105, 241-T-102, 241-C-104, and 241-BX-109 are to be analyzed using Module A in the Tank Waste Remediation System Tank Waste Characterization Plan (Reference 4). The core from Tank 241-C-105 will be analyzed using Module F. Table 2 lists the additional samples that will be obtained and the analytical modules to be used.

Table 2

Tank #	SST/DST	# Cores	# Segments/ Core	Type of Tank	Module
241-T-105	SST	2	2	Non-Watch List	A
241-T-102	SST	2	1	Non-Watch List	A
241-C-105	SST	1	3	Early Feed	F
241-C-104	SST	2	6	Non-Watch List	A
241-BX-109	SST	2	4	Non-Watch List	A

The determination of which core samples will be sent to the 325 Laboratory for analysis will be made by Hanford Analytical Services Management before the sampling of that core begins. The following tanks are currently planned to be sent directly to the 325 Laboratory: 241-T-102, 241-C-106, 241-C-105, 241-C-108, 241-BX-109, and 241-C-103. It is expected that portions of the other core samples will be sent from the 222-S laboratory for selected analyses. The 325 Laboratory is authorized to receive and extrude these samples, photograph the segments, perform the measurements that must be done before the samples are subdivided and placed into jars, obtain any necessary unhomogenized subsamples, and store the samples until the TPP is approved. Homogenization, compositing, and analyses are to be performed only after the TPP is approved.

This change has been determined to be WHC impact level 4. This LOI will be added to the SOW in an appendix and the table of contents of the SOW will be updated. Should there be any exceptions to this LOI which are not already documented in the draft laboratory technical project plan (Reference 1), the laboratory is requested to provide written notification to Analytical Customer Interface and Analytical Evaluation and Reporting by March 31, 1993. Work order number ED3540 for $250K is attached for initiation of work on SST core samples taken in FY 1993. If there are any questions, please contact Mr. K. L. Silvers at 372-2485 or Ms. L. M. Sasaki at 373-1027.

K. L. Silvers, Acting Manager
Analytical Customer Interface
Hanford Analytical Services Management

A. F. Noonan, Manager
Analytical Evaluation and Reporting

pkc

Attachment

PNL - R. M. Bean
W. B. Gintner
S. G. McKinley
V. P. Ostrander

ED3540 -
WORK ORDER

54-30 58 '01 991

*SERVICING/ COST CENTER	*CUSTOMER/ SUPPORT CODE	SUB-ACCOUNT/ ACCT. CLASS	REF NO WWO	AUTH. FUNDS	AREA	BLDG.	(1)
D	N435R			$250000	300	3225	

START DATE	TERM DATE	OVERHEAD CODES	PROJECT NO.
030193	093093		

RESPONSIBLE ORG.	QA LEVEL	QA REVIEW	FY FUNDS	PR. NO.	SYSTEM
W7K220	30	T. F. Whelan	1993		

DESCRIPTION	FACILITIES CHANGE NOTICE - ☐ YES ☐ NO	ASSIGNED TO	FM. NO.
SST Core Sample Analysis - Initiate work on SST cores per SOW (WHC-SOW-93-0002) and Correspondence #9352556 (letter of instruction)		S. G. McKinley	
		ESTIMATED COST (INCLUDES REFERRAL ORDERS)	
		HOURS	
		LABOR & TIME	
		MATERIAL	
		OTHER	
		TOTAL	$250,000

BUDGET APPROVAL

BUDGET APPROVAL	DATE		LINE OR COST ACCOUNT
A. F. Noonan C. M. Welch	3/29/93		

ISSUED BY	DATE	PHONE	ACCEPTED BY	ASSIGNED SERV.
L. M. Sasaki	3-25-93	373-1027		D

APPROVED BY	DATE	COMPLETED - FMN	DATE	ISSUING ORGAN
J. G. Tropson	3-29-93			W7K220

* FIRST DIGIT COMPANY INDICATOR: D - PNL R - KEH M - HEHF W - WHC 9 - DOE (1) FOR INTERNAL CONTRACTOR USE ONLY.

A U.S. GOVERNMENT PRINTING OFFICE: 1992—683 514

P.O. Box 1970 Richland, WA 99352

July 13, 1993

9355888

Mr. A. G. King, Manager
Analytical Chemistry Laboratory
Pacific Northwest Laboratory
Post Office Box 999
Richland, Washington 99352

Dear Mr. King:

LETTER OF INSTRUCTION PROVIDING GUIDANCE AND PRIORITY FOR SAMPLE ANALYSIS OF TANK T-102 CORES 55 AND 56

References: (1) WHC-SD-WM-PLN-047, Revision 0, "Tank Waste Remediation System Tank Waste Characterization Plan," dated December 1992.

(2) WHC-SOW-93-0002, Revision 0, "Sampling and Analysis of SST and DST Waste Tanks in Support of TWRS Fiscal Year 1993," dated November 1992.

This letter of instruction (LOI) provides guidance and analytical priority to the 325 Laboratory for the characterization of Single-Shell Tank 241-T-102 cores 55 and 56.

Because of a shortage of sample material, the complete suite of analysis for Module A cannot be conducted on each core. As a result, the analysis will be prioritized for the tank on a core-by-core bases. Sample material from core 56 will be recovered and stored until further notice. The table below provides the analysis and subsequent priority for core 55 sample material. The analysis shall be conducted in the order of priority until there is no longer any sample remaining.

Table - Priorities for Analysis of Solids Core Composite

Priority	Analysis	Initial* or Duplicate Sample
1	bulk density	initial
2	water leach: IC, nitrite	initial/duplicate
3	acid leach and fusion: ICP	initial
4	fusion: radionuclides (GEA, Pu239/240, Am241, Sr90, total U, Pu/U isotopic, Tc99, total alpha, total beta)	initial
5	water leach: ammonia, TOC, carbonate, ICP, GEA, C14, H3, total alpha, total beta	initial
6	direct: CN, TOC, DSC/TGA	initial

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WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

Priority	Analysis	Initial* or Duplicate Sample
7	wt% solids, wt % oxides	initial
8	water leach: Cr(VI), TDS water leach residual solids: dry weight, GEA	initial
9	acid leach and fusion: ICP	duplicate
10	fusion: radionuclides (GEA, Pu239/240, Am241, Sr90, total U, Pu/U isotopic, Tc99, total alpha, total beta)	duplicate
11	water leach: ammonia, TOC, carbonate, ICP, GEA, C14, H3, total alpha, total beta	duplicate
12	direct: CN, TOC, DSC/TGA	duplicate
13	wt% solids, wt% oxides	duplicate
14	water leach: Cr(VI), TDS water leach residual solids: dry weight, GEA	initial/duplicate
15	pH, OH	initial/duplicate
16	Hg, I129	initial/duplicate
17	rheology, PSA (unhomogenized samples)	initial
18 (due to lack of method)	ICP/MS (I129, noble metals), TCO	initial/duplicate

* "initial" to include spikes if required

This LOI providing analytical guidance has been determined to be Impact Level 4. If you have any questions, please contact Ms. L. M. Sasaki at 373-1027 or Ms. H. S. Rich at 372-2485.

Very truly yours,

L. M. Sasaki

L. M. Sasaki, Engineer
Analytical Evaluating and Reporting

H. S. Rich, Scientist
Analytical Customer Interface

jgi

PNL - S. G. McKinley
S. A. Schubert
J. M. Tingey

JA-162

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CHAIN-OF-CUSTODY RECORD FOR CORE SAMPLING

(1) Shipment Number 593-008 (2) Sample Number 93-009 (3) Supervisor* D. Hartley 4/1/93

(4) Tank 102T (5) Riser 2 (6) Segment 1 (7) Core 055 (8) Cask Serial Number C-100-T-400C

Radiation Survey Data:	(9) FIELD	(26) LABORATORY
Over Top Dose Rate	<1.5MR/HR	<0.5 mr/hr
Side Dose Rate	<1.5MR/HR	<0.5 mr/hr
Bottom Dose Rate	<1.5MR/HR	<0.6 mr/hr
Smearable Contamination	<Det (alpha)	<D (alpha)
	<Det (beta-gamma)	<D (beta-gamma)
	RPT* (Signature)	RPT* (Signature)

(10) Shipment Description:

A. Work Package Number	2W-92-01156-W
B. Cask Seal Number	4359
C. Sampler Number Used	91-148
D. Date and Time Sampler Unseated	3-25-93, 0332
E. Expected Liquid Content	20%
F. Expected Solid Content	80%
G. Dose Rate Through Drill String	120MR/HR
H. Expected Sample Length	7'

(11) INFORMATION *(Include statement of laboratory tests to be performed.)*

WHC-SD-WM-PLN-047, Module A

(12) Field Comments:

(27) Laboratory Comments

(13) POINT OF ORIGIN	(14) TENDER NAME	(16) DATE RELEASED	(18) DESTINATION	(20) RECIPIENT NAME	(22) DATE RECEIVED
241-T-102	D. Hartley	4-1-93	325 300 Area	JK Andrews	4-1-93
	(15) TENDER SIGNATURE* D Hartley	(17) TIME RELEASED 1930		(21) RECIPIENT SIGNATURE* JK Andrews	(23) TIME RECEIVED 2145

(19) Seal Intact Upon Release?	(24) Seal Intact Upon Receipt?	(25) Seal Data Consistent with this Record?		
		Shipment No.	Cask Seal No	Sample No.
☐ Yes ☐ No	☑ Yes ☐ No	☑ Yes ☐ No	☑ Yes ☐ No	☑ Yes ☐ No

DISTRIBUTION: White – Office of Sample Management; Pink – Process Engineering, R1-51; Yellow – Recipient of Sample; Goldenrod – Tank Farm Operations, T4-01

BC-6000 309 (02/90)

WHC-SD-WM-DP-052-
ADDENDUM 1 REV. 0

CHAIN-OF-CUSTODY RECORD FOR CORE SAMPLING

(1) Shipment Number 593-008 (2) Sample Number N/A (3) Supervisor* D.H. HARTLEY

(4) Tank 102T (5) Riser N/A (6) Segment N/A (7) Core 055/056 (8) Cask Serial Number C1033

Radiation Survey Data:	(9) FIELD	(26) LABORATORY
Over Top Dose Rate	<6 mR/Hr	<0.5 mR/h
Side Dose Rate	<1 mR/Hr	<0.5 mR/h
Bottom Dose Rate	<1 mR/Hr	<0.5 mR/h
Smearable Contamination	<Det. (alpha)	<D.L (alpha)
	<Det. (beta-gamma)	<D.L (beta-gamma)
	RPT* (Signature)	RPT* (Signature)

(10) Shipment Description:

A. Work Package Number	2W-92-01156-W
B. Cask Seal Number	4358
C. Sampler Number Used	91-118
D. Date and Time Sampler Unseated	3-28-93, 1015
E. Expected Liquid Content	100%
F. Expected Solid Content	0
G. Dose Rate Through Drill String	N/A
H. Expected Sample Length	191"

(11) INFORMATION *(Include statement of laboratory tests to be performed.)*

WHC-SD-WM-PLN-047, Module A
WATER SAMPLE FOR 102T

(12) Field Comments:

(27) Laboratory Comments

(13) POINT OF ORIGIN	(14) SENDER NAME	(16) DATE RELEASED	(18) DESTINATION	(20) RECIPIENT NAME	(22) DATE RECEIVED
241-T-102	D. Hartley	4-1-93	225 300 Area	TK Andrews	4-1-93
	(15) SENDER SIGNATURE* D.H. Hartley	(17) TIME RELEASED 1930		(21) RECIPIENT SIGNATURE* TK Andrews	(23) TIME RECEIVED 2145

(19) Seal Intact Upon Release?	(24) Seal Intact Upon Receipt?	(25) Seal Data Consistent with this Record?		
		Shipment No.	Cask Seal No.	Sample No.
☑ Yes ☐ No	☑ Yes ☐ No	☑ Yes ☐ No	☑ Yes ☐ No	☑ Yes ☐ No

DISTRIBUTION: White – Office of Sample Management; Pink – Process Engineering, R1-51; Yellow – Recipient of Sample; Goldenrod – Tank Farm Operations, T4-01

BC-6000-309 (02/90)

WHC-SD-WM-DP-052-
ADDENDUM 1 REV. 0

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B02-003

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CHAIN-OF-CUSTODY RECORD FOR CORE SAMPLING

(1) Shipment Number 593-008 (2) Sample Number 93-010 (3) Supervisor* D. Hartley

(4) Tank 102T (5) Riser 8 (6) Segment 1 (7) Core 056 (8) Cask Serial Number C1032

Radiation Survey Data:	(9) FIELD	(26) LABORATORY
Over Top Dose Rate	1 mV/hr	<0.5 mR/h
Side Dose Rate	2.0 mV/hr	2.0 mR/h
Bottom Dose Rate	2.5 mV/hr	2.5 mR/h
Smearable Contamination	<100 (alpha)	<DL (alpha)
	<100 (beta-gamma)	<DL (beta-gamma)
	RPT* (Signature)	RPT* (Signature)

(10) Shipment Description:

A. Work Package Number	241-92-01156-W
B. Cask Seal Number	4360
C. Sampler Number Used	91-020
D. Date and Time Sampler Unseated	3-26-93, 0939
E. Expected Liquid Content	20%
F. Expected Solid Content	80%
G. Dose Rate Through Drill String	40 mV/hr
H. Expected Sample Length	7"

(11) INFORMATION *(Include statement of laboratory tests to be performed.)*

WHC-SD-WM-PLN-047 Module A

(12) Field Comments:

(27) Laboratory Comments

(13) POINT OF ORIGIN	(14) SENDER NAME	(16) DATE RELEASED	(18) DESTINATION	(20) RECIPIENT NAME	(22) DATE RECEIVED
241-T-102	D. Hartley	4-1-93	325 300 Area	JK Andrews	4-1-93
	(15) SENDER SIGNATURE*	(17) TIME RELEASED		(21) RECIPIENT SIGNATURE*	(23) TIME RECEIVED
	D. Hartley	1930		JK Andrews	2154

(19) Seal Intact Upon Release?	(24) Seal Intact Upon Receipt?	(25) Seal Data Consistent with this Record?		
		Shipment No.	Cask Seal No.	Sample No.
☐ Yes ☐ No	☑ Yes ☐ No	☑ Yes ☐ No	☑ Yes ☐ No	☑ Yes ☐ No

DISTRIBUTION: White – Office of Sample Management; Pink – Process Engineering, R1-51; Yellow – Recipient of Sample; Goldenrod – Tank Farm Operations, T4-01

BC-6000-309 (02/90)

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WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

CHAIN-OF-CUSTODY RECORD FOR CORE SAMPLING

(1) Shipment Number S93-009 (2) Sample Number 93-009 (3) Supervisor* M.C. JONES

(4) Tank 102T (5) Riser 2 (6) Segment 1 (7) Core 055 (8) Cask Serial Number 91-148 1004C

Radiation Survey Data:	(9) FIELD	(26) LABORATORY
Over Top Dose Rate	< 1	< 1
Side Dose Rate	< 1.5	< 1.5
Bottom Dose Rate	2.0	2.0
Smearable Contamination	< Det (alpha)	< D (alpha)
	< Det (beta-gamma)	< D (beta-gamma)
	RPT* [signature]	RPT* [signature]

(10) Shipment Description:

A. Work Package Number	2W-92-01156-W
B. Cask Seal Number	1004C 4357
C. Sampler Number Used	4357 91-148
D. Date and Time Sampler Unseated	3-25-93, 10:32
E. Expected Liquid Content	20%
F. Expected Solid Content	80%
G. Dose Rate Through Drill String	120 mr/HR
H. Expected Sample Length	7"

(11) INFORMATION *(Include statement of laboratory tests to be performed.)*

WHC-SD-WM-PLN-047, Module A

(12) Field Comments:

This sample was the one that should have been sent on 4-1-93 with sample 93-010. It has been in SX Tank Farm for the last month.

(27) Laboratory Comments

(13) POINT OF ORIGIN	(14) SENDER NAME	(15) SENDER SIGNATURE*	(16) DATE RELEASED	(17) TIME RELEASED	(18) DESTINATION	(20) RECIPIENT NAME	(21) RECIPIENT SIGNATURE*	(22) DATE RECEIVED	(23) TIME RECEIVED
241-SX	M.C. JONES	[signature]	5-4-93	6:00 P.M.	325 300 Area	TK ANDREWS	[signature]	5-4-93	2052

(19) Seal Intact Upon Release?	(24) Seal Intact Upon Receipt?	(25) Seal Data Consistent with this Record?		
		Shipment No.	Cask Seal No.	Sample No.
☒ Yes ☐ No	☒ Yes ☐ No	☐ Yes ☐ No	☒ Yes ☐ No	☐ Yes ☐ No

DISTRIBUTION: White – Office of Sample Management; Yellow – Recipient of Sample; Pink – Process Engineering, R1-51; Goldenrod – Tank Farm Operations, T4-01

BC-6000-309 (02/90)

WHC-SD-WM-DP-052.
ADDENDUM 1 REV. 0

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B02-005

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SUPPLEMENTAL INFORMATION

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WHC-SD-WM-DP-052-
ADDENDUM 1 REV. 0

DEFICIENCY REPORT

QA Plan ALO-003, R1
Program TWC
Task 20777
File Cat. Q6

1. DR Number:	2. Date:	3. QA Plan Number:	4. Impact Level:	5. Project/Activity Number:	6. Org. Code:
93-031	7/19/93	ALO-003, Rev. 1	II	20777	D7E15

7. PROJECT/ACTIVITY TITLE: Tank Waste Characterization Project

7a. CLIENT NAME: WHC

8. REQUIREMENT AND SOURCE OF REQUIREMENT (Document No., Revision, and Title):

PNL-MA-599 Manual, Procedure #PNL-ALO-051; Sample Receiving
Section 3.0 states in part "The following information is recorded on the sample receipt form (Exhibit 1): ...Verification that the sample identification agrees with chain-of-custody document"

9. DESCRIPTION OF DEFICIENCY:

The Chain-of-Custody (COC) was signed when samples were received and the Sample Receipt Form was prepared. The samples were received within the Shielded Analytical Laboratory (SAL) in labelled cans. The COC and Sample Receipt Form were consistent with the identifications on the sample cans. The samples were removed from the cans approximately one month later. The labelling on the samples within the cans were not consistent with that of the COC. WHC sample #D49 was missing this identification on the sample bottle and the labelling indicated the sample was "liner liquid" but it was a solid material. The Sample Receipt Form did not reflect these discrepancies.

9a. CORRECTIVE ACTION RESPONSE DUE: N/A

10. NCR Required? NO [ ] YES [X]	10a. Referenced Surveillance Number or Other	11.
Number: PNL-93-038	Number: N/A	Richard L. Fuller 8/16/93 Originator's Signature and Date

12a. EVALUATION AND CORRECTIVE ACTION (Cause of Deficiency):

The samples are not removed from the cans until they can be processed. This is done for several reasons: to keep the radiation exposure ALARA, sample analysis schedule may not have been established, and sample material may need to be transferred to another group so that the sample container may be opened in the most appropriate location. Due to these reasons, the Sample Receipt Form is prepared without verifying the content of the sample container.

12b. Effect of Deficiency on Validity and Integrity of Results:

[X] None [ ] Unknown [ ] Some [ ] Significant

Explain: Sample identification was verified when cans were opened. Discrepancy was noted and resolved (see 12f. below).

12c. If Unknown, Some or Significant effect, (Identify results affected or potentially affected):

12d. ACTION TO CORRECT DEFICIENCY: NONE

12e. ACTION TO PRECLUDE RECURRENCE:

Procedure PNL-ALO-051 will be revised to incorporate the receipt of sample containers and what actions are necessary when the samples are removed from the container.

Expected Completion Date: September 24, 1993

12f. CONTACT CLIENT? [ ] No [X] Yes—
Contacted (Name and date), Contacted by (Name and Date): Contacted Keith Fuller, WHC, 7/13/93 by SG McKinley, ACL Project Manager

Explain: One of four samples were received with improper labelling, therefore samples were matched with that of the COC and the incorrect sample was determined to be sample D49 by elimination. The sample was also labelled as "liner liquid" which was incorrect. Discussion with Keith Fuller further verified that the incorrectly labelled sample was indeed the sample as stated on the COC.

DEFICIENCY REPORT

DR NUMBER 93-033

12d. ACTION TO CORRECT DEFICIENCY:

Actions to correct deficiencies are intermediate actions to allow characterization analyses to continue. Actions to achieve compliance with QA Plan ALO-003 are detailed in 12E.

1) Cs analyses will be conducted by utilizing the Bi GFAA procedure (PNL-ALO-216) with minor modifications documented in the data package.
2) If CN analyses utilize the autoanalyzer colorimetric finish, the vendor's analytical procedure will be used and documented in the data package.
3) The ICP will continue to operate under DR-91-089; concentrations of all standards (e.g., cal, ICS, verify) are documented in system file.
4) The mercury analysis will continue to operate under DR-91-099; modifications to PNL-ALO-213 will be documented in the data package.
5) Where appropriate MDLs will be established by QA Plan recommendation; alternate methods will be documented in the data package.
6) The current acid "SST spiking solution" will be used in lieu of the acid-based spiking solution required by the QA Plan (B, Ce, Cs, La, Li, Mg, Mo, Nd, P, Sr and Ti are not in the current spiking solution)
7) The current "SST metals and anion spiking solutions" will be used in lieu of the spiking solution(s) required by the QA Plan (B, Ce, Cs, La, Li, Mg, Mo, Nd, P, Sr, Ti, NH3, Cr(VI), are not in the current spiking solutions)
8) A CLP CRI standard will be used in lieu of the CRI standard required by the QA Plan.
9) The current "SST ICS" will be used in lieu of the ICS required by the QA Plan; additional high concentration interferences shall be evaluated by using single analyte standards.

12E. ACTION TO PRECLUDE RECURRENCE:

The single action to preclude recurrence is to upgrade procedures and standards to fully support the intent of the QA Plan. The individual activities are summarized below. Additional details, wherever necessary, will be documented in the Tank Waste Characterization Project file.

1) Both a Cs Flame AA and GFAA procedure will be written, approved, and included in PNL-MA-599.
   Expected Completion Date: Nov 1, 1993
2) Procedures (i.e., midi-distillation, autoanalyzer, titration) to support the CN analysis will be written, approved, and included in PNL-MA-599.
   Expected Completion Date: Nov 1, 1993
3) Revision to the ICP procedure to be completed, approved, and included in PNL-MA-599.
   Expected Completion Date: Nov 1, 1993
4) Revision to the mercury procedure to be completed, approved, and included in PNL-MA-599.
   Expected Completion Date: Nov 1, 1993
5) Where appropriate, "instrument specific" procedures for establishing IDLs/MDLs will be developed and submitted for approval.
   Expected Completion Date: Sept 30, 1993
6) Where compatible, pre/post spiking solutions, based on historical data, will be developed which fully support the QA Plan.
   Expected Completion Date: Oct 31, 1993
7) Water-based (or very low acid) spiking solutions based on historical water leach analytes present will be developed.
   Expected Completion Date: Nov 1, 1993
8) "Instrument specific" MDL check standards will be developed which fully support the QA Plan.
   Expected Completion Date: Nov 1, 1993
9) "Tank characterization" ICP Interference Check Standards based on historical A/B/C analytes will be developed.
   Expected Completion Date: Oct 31, 1993

12f. CONTACT CLIENT? [ ] No [X] Yes
Contacted (Name and date), Contacted by (Name and Date):

Explain:

12g. SA Schubert for SAS 8/6/93
Cognizant Manager Signature and Date

12h. TL Ehlert 8/6/93
PQD Representative Signature and Date

13a. Criteria No.(s): 11	13b. Def. Code(s): A3	13c. Cause Code(s): O	13d. SCL(s): III

14. Corrective Action Complete/Comments:

Closeout: _____
PQD Representative Signature/Date

15. Distribution:

Cognizant Mgr:	SA Schubert
Project/Activity Mgr.:	SG McKinley
Originator:	MW Urie
QS&R:	LL Arel
PQD Rep.:	TL Ehlert
Others:	AG King
	KJ Kuhl-Klinger

IA-174
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WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

DEFICIENCY REPORT

QA Plan ALO-003, R1
Program TWC
Task 20777
File Cat QA

1. DR Number: DR-93-033	2. Date: 7/23/93	3. QA Plan Number: ALO-003, Rev. 1	4. Impact Level: II	5. Project/Activity Number: 20777	6. Org. Code: D7E15

7. PROJECT/ACTIVITY TITLE: Tank Waste Characterization Project

7a. CLIENT NAME: WHC

8. REQUIREMENT AND SOURCE OF REQUIREMENT (Document No., Revision, and Title):

PNL-ALO-003, Rev 1: QA Plan for Analytical Activities in M&CS Center in support of TWCP

9. DESCRIPTION OF DEFICIENCY:

The QA Plan (ALO-003) has been approved and is the controlling document for conducting analytical work within ACL. The first tank to be analyzed under this plan is ready for processing and there has been insufficient time to implement all the changes in analytical operations required by the this new QA Plan. Also, there is only a small quantity of tank material for testing and establishing of operational protocols and standard levels and there is not sufficiet material to repeat the analyses. Areas most affected, and for which deficiencies have been noted, are the requirement for additional multi-analytes standards with specific analytes (e.g., pre and post spiking solutions, interference check standards based on tank compositions, and specific MDL check standards), the need for procedures (or modifications to existing procedures), and the need to find alternate ways to establish IDLs and MDLs.

9a. CORRECTIVE ACTION RESPONSE DUE:

10. NCR Required? NO [X] YES [ ] Number:	10a. Referenced Surveillance Number or Other Number: N/A	11. M W Winn 8/2/93 Originator's Signature and Date

12a. EVALUATION AND CORRECTIVE ACTION (Cause of Deficiency):

Based on the current turn around times required for the analytical work on the current SST Tanks in the ACL, the requirements within the governing QA Plan can not be implemented in time to support the analytical work. The following issues have been identified:

1) Approved PNL procedure is not available to support Cs (by Flame AA or GFAA).
2) Approved PNL procedure is not available to support Total CN analysis by new autoanalyzer system.
3) ICP procedure is currently under a previous DR; procedure revision is in progress.
4) Hg procedure is currently under a previous DR; procedure revision is in progress.
5) Difficulties are being experienced in establishing IDL/MDL per QA Plan; QA Plan method is inappropriate for some analyses.
6) Pre/Post spiking solutions as defined by the QA Plan are not ready for use; require preparation/verification prior to use.
7) Water Leach spiking solutions are not available at this time.
8) CRI standards addressing all analytes of interest are not available at this time.
9) Interference check standards based on tank characterization have not been prepared, verified, or tested for stability.

12b. Effect of Deficiency on Validity and Integrity of Results:

[ ] None [X] Unknown [ ] Some [ ] Significant

Explain:
Analytical procedures are essentially self-qualifying; however, lack of some standards at specific concentrations (e.g., ICS) has an unknown effect.

12c. If Unknown, Some or Significant effect, (Identify results affected or potentially affected):

The analytical results for the ICP and AA at or near the MDL may be bias. Lack of an MDL standards covering all analytes, spiking solutions to evaluate all A/B analytes, and a "tank characterization" ICS standard for evaluating interference corrections, potentially affect the "accurate" quantitation of the analytes of interest.

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

DEFICIENCY REPORT

DR NUMBER 93-031

12g. S.G. McKinley 8/17/93 Cognizant Manager Signature and Date	12h. TL Eckert 8/16/93 PQD Representative Signature and Date

13a. Criteria No.(s): 15	13b. Def. Code(s): A3	13c. Cause Code(s): C	13d. SCL(s): IV

14. Corrective Action Complete/Comments:

Closeout _____
PQD Representative Signature/Date

15. Distribution:

Cognizant Mgr: SG McKinley — Project/Activity Mgr: SG McKinley
Originator: RT Steele — QS&R: LL Arel
PQD Rep.: TL Eckert — Other: AG King

IA-176

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DEFICIENCY REPORT

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

1. DR Number: DR-93-041	2. Date: 09/15/93	3. QA Plan Number: ALO-003, Rev. 1	4. Impact Level: II	5. Project/Activity Number: 20777	6. Org. Code: D7E15

7. PROJECT/ACTIVITY TITLE: Tank Waste Characterization Project

7a. CLIENT NAME: WHC

8. REQUIREMENT AND SOURCE OF REQUIREMENT (Document No., Revision, and Title):

PNL-ALO-003, Rev. 1, Section 11.0: QA Plan for Analytical Activities in M&CS Center in Support of TWCP.

9. DESCRIPTION OF DEFICIENCY:

The QA Plan (ALO-003, Rev. 1) has been approved and is the controlling document for conducting analytical work within the ACL. The plan specifies that an ICV, ICB shall be run at the beginning of the run (after calibration) and a CCB/CCV shall be run every ten samples thereafter and after the last analytical sample. Results generated during the analysis of Cr^{6+} did not meet this requirement.

9a. CORRECTIVE ACTION RESPONSE DUE:

10. NCR Required? NO [X] YES [ ] Number:	10a. Referenced Surveillance Number or Other Number: N/A	11. Originator's Signature and Date: 9/15/93

12a. EVALUATION AND CORRECTIVE ACTION (Cause of Deficiency):

Analyst and Cognizant Scientist overlooked the requirement.

12b. Effect of Deficiency on Validity and Integrity of Results:

[X] None [ ] Unknown [ ] Some [ ] Significant

Explain: A spike sample was run at the end of the analysis and a good recovery (106%) was obtained. This indicates the procedure was in control during the analysis.

12c. If Unknown, Some or Significant effect, (Identify results affected or potentially affected): N/A

12d. ACTION TO CORRECT DEFICIENCY:

Review QC Requirements with personnel and conform in future campaigns.

12e. ACTION TO PRECLUDE RECURRENCE: Expected Completion Date:

Cognizant Scientist review data upon completion of analysis.

12f. CONTACT CLIENT? [X] No [ ] Yes
Contacted (Name and date), Contacted by (Name and Date):

Explain:

12g. Cognizant Manager Signature and Date: 9/17/93	12h. POD Representative Signature and Date: for T R Ehlert 9-17-93

13a. Criteria No.(s): 11	13b. Def. Code(s): C4	13c. Cause Code(s): A	13d. SCL(s): III

IA-177

A02-006

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DEFICIENCY REPORT

DR NUMBER 93-041

14. Corrective Action Complete/Comments:	15. Distribution:
Closeout: _____ PQD Representative Signature/Date	Cognizant Mgr: Project/Activity Mgr.: Originator: QS&R: PQD Rep.:

A3 - ACL DEVIATION REPORTS

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ADR Number: RTS 9/3/93 1

ANALYTICAL CHEMISTRY LABORATORY
DEVIATION REPORT

INITIATED BY: Rick T. STEELE DATE: 9/3/93

REQUIREMENTS DOCUMENT: PNL-ALO-482 Rev. 1, ALO-003 Rev. 1

TYPE OF DEVIATION:	INFORMATION AFFECTED:	CHANGE RECOMMENDATION:
X PREPLANNED	___ CRITICAL	___ TEMPORARY
___ REAL-TIME	___ SIGNIFICANT	X PERMANENT

PROJECTS AFFECTED:	SAMPLES AFFECTED:
20777	93-08755-C1, 93-08755-C2, 93-08755-C3
	93-08755-C4

DEVIATION DESCRIPTION: Pre Digestion spike for C-14 was completed AND Not the post Digestion spike called for by the New QAPjP ALO-003 Rev. 1

EXPECTED IMPACT: NONE

RESULTING ACTIONS: The QAPjP is incorrect AND needs to be changed. This revision will be an enhancement to the data quality as it show recovery in the matrix. The QAPjP will be updated to reflect this change.

APPROVAL: Rick T. Steele DATE: 9/3/93
Technical Group Leader

CONCURRENCE/APPROVAL: [signature] DATE: 9/7/93 (If Project Specific)
Project Manager

Distribution:
Required: TGL Procedure Coordinator
As Appropriate: PM Project/System File Other: _____

pg 1 of 1

ADR693.TMP

ADR Number: RTS 9/3/93 2

ANALYTICAL CHEMISTRY LABORATORY
DEVIATION REPORT

INITIATED BY: R T STEELE DATE: 9/3/93

REQUIREMENTS DOCUMENT: TI-TWRC-06[illegible]; PNL-ALO-504 Rev.1

TYPE OF DEVIATION:	INFORMATION AFFECTED:	CHANGE RECOMMENDATION:
X PREPLANNED	___ CRITICAL	___ TEMPORARY
___ REAL-TIME	___ SIGNIFICANT	X PERMANENT

PROJECTS AFFECTED:	SAMPLES AFFECTED:
20799	93-08755-K-1, 93-08755-K-2

DEVIATION DESCRIPTION: WRONG, i.e. ANOTHER FORM, WAS USED TO document the results.

EXPECTED IMPACT: NONE

RESULTING ACTIONS: PNL-ALO-504 Needs to be revised so that the form in Exhibit 7 is recommended AND NOT MANDATORY.

APPROVAL: Richard T Steele DATE: 9/3/93
Technical Group Leader

CONCURRENCE/APPROVAL: [signature] DATE: 9/7/93 (If Project Specific)
Project Manager

Distribution:
Required: TGL Procedure Coordinator
As Appropriate: PM Project/System File Other: ___

pg 1 of 1

ADR693.THP

ADR Number: RTS 9/24/93

ANALYTICAL CHEMISTRY LABORATORY
DEVIATION REPORT

INITIATED BY: RT Steele DATE: 9/24/93

REQUIREMENTS DOCUMENT: QAPjP AL0-003 Rev 1

TYPE OF DEVIATION:
- ✓ PREPLANNED
- ___ REAL-TIME

INFORMATION AFFECTED: NA
- ___ CRITICAL
- ___ SIGNIFICANT

CHANGE RECOMMENDATION:
- ___ TEMPORARY
- ✓ PERMANENT

PROJECTS AFFECTED:	SAMPLES AFFECTED:
TWC 20777	see attached pages from T-102 Data Report

DEVIATION DESCRIPTION: see attached

EXPECTED IMPACT: none

RESULTING ACTIONS: revise sample preparation procedures

APPROVAL: Robert T. Steele (Technical Group Leader) DATE: 9/24/93

CONCURRENCE/APPROVAL: [signature] (Project Manager) DATE: 9/24/93 (If Project Specific)

Distribution:
Required: TGL Procedure Coordinator
As Appropriate: PM Project/System File Other:

pg ___ of ___

ADR693.THP

IA 182

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Sample preparations/distributions of the blended Core 55 solids involved:

1) Water leaching for IC, ICP, NH_3, TOC/TIC/TC, Cr(VI), TDS, pH, OH, GEA, Total Alpha, Total Beta, C-14, H-3.

2) Ni/KOH fusions for ICP, GEA, Total Alpha, Total Beta, AEA for Pu,Np,Am, Uranium, Tc-99, Sr/Y-90, and Pu/U isotopics.

3) Acid digestions or distillations for ICP, CN and Hg.

4) The distribution of direct sub-samples for DSC/TGA and Weight Percent Oxides.

Following the water leaching process, the undissolved sample residue was dried and weighed. Portions of the dried solids were fused (Ni/KOH) for GEA.

Predigestion spikes were performed for ICP metals, IC anions, cyanide, carbon, C-14 and mercury analyses only. Post digestion spiking was done at the laboratory bench by the functional group performing the analysis.

Bulk Density, Weight Percent and Total Dissolved Solids determinations were completed in-cell. A 1:5 (not 1:1) water contact was made for pH and OH^- due to limited sample availability.

The Tank T-102 work scope included both a Field and Hot Cell Blank. Per Test Instruction, portions of each blank were acidified with nitric acid and distributed to the laboratories for ICP, NH_3, GEA, Total Alpha and Total Beta. A portion of each blank was filtered (0.45 micron) for ion chromatography. Direct blank portions were distributed for TOC, CN, pH, OH and DSC/TGA analyses.

During sample preparation, the SAL made deliberate minor deviations to sample preparatory procedures for one or more of the following reasons:

ADR
RTS 9/24/93

1) Insufficient sample was available to conduct the analyses per procedure while maintaining the level of quality control requested.

2) Sample weights and/or final volumes were reduced to facilitate waste minimization.

3) Sample weights and/or final volumes were altered to increase the concentration of certain analytes of interest. This was done to meet the procedural concentration ranges needed to perform the analyses.

Sample sizes and final volumes for all sample preparations are documented on the Sample Preparation Sheets included in Appendix B5. Table 1-3 lists the sample preparatory procedure deviations performed during the processing of Tank T-102.

Table 1-3: T-102 Core 55, Sample Preparation Procedural Deviations

ALO Number	Prep Method	Sample Size Deviation	Sample Volume Deviation	Reagent Deviation	Observed Effect
93-08755-A	Acid	Yes	No	No	None
93-08755-C	Water	Yes	Yes	No	None
93-08755-D	Acid (Hg)	No	Yes	No	None
93-08755-G	Acid (CN)	Yes	Yes	No	None
93-08755-K	Wt% Solids	Yes	N/A	N/A	None
93-08755-N	Water	Yes	Yes	N/A	None

ADR
RTS 9/24/96

THERMAL ANALYSIS

Differential Scanning Calorimetry (DSC) and Scanning Thermogravimetry (STG) were performed in duplicate on the unhomogenized material from Core 55. DSC and STG were also performed on the field blank, hot cell blank, and the water used to prepare the hot cell blank. These two thermal analysis techniques are useful in determining the thermal stability and reactivity of a material. DSC measures heat released or absorbed while the temperature of the sample is increased at a constant rate. Data generated by the DSC analysis is often used to measure thermal decomposition temperatures, heats of reaction, reaction temperatures, melting points and solid-solid transition temperatures. STG measures the mass of a sample while the temperature of the sample is increased at a constant rate. The STG data is used to measure thermal decomposition temperatures, water

DATE TO QC: August 17, 1993

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - Bulk Density

Data Package/Report - Core 55

ACL Numbers - 93-08755

Kristine J. Kuhl-Klinger
PNL ACL Quality Representative

8/25/93
Date

14-185

C04-002

000173

DATE TO QC: August 16, 1993

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - Particle Size

Data Package/Report - Core 57 (Segments 1 & 2) 5 sets 9/23/93

ACL Numbers - 93-07987-M1 93-07987-M2 93-07988-M1 93-07988-M2

Kristine J. Kuhl-Klinger
PNL ACL Quality Representative

9/25/93
Date

IA- 186

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Battelle
Pacific Northwest Laboratories

Project Number _____

Internal Distribution

Date August 12,1993

To SG McKinley

From MC Burt

Subject Particle Size Distribution of SST Samples

Particle size distribution analysis by procedure PNL-ALO-599-530 was performed on the samples from Tank T-102, Core 55 and designated 93-10374-M1 and 93-10374M2. M & TE used was the Brinkmann 2010 Particle Size Analyzer, WC02018. A glass sphere reference standard, Duke 147, at a nominal MPD (mean particle diameter) of 20 μm is measured with each sample batch. A volume density distribution plot is provided for the standard with both volume and number density plots and additionally number and volume distribution tables (ranges) included for the samples.

The samples as received from the Hot Cells were a very light colored, fine, dry powder. Approximately 2 mL of 50% glycerol mixture was added to both moisten and suspend the sample particles.

As viewed on the system monitor the samples were well dispersed and no large or agglomerated particles were observed. Because particles larger than 60 μm were present the samples were measured in the 'Regular' mode which measures particles in the range of 0.5 to 150 microns.

If there are questions regarding the results please contact MC Burt on 376-3762.

MC Burt, Sr. Res.Scientist
Analytical Chemistry Laboratory

Concur: 8/12/93

PART 930812

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C05-003

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GALAI — CIS-1
Computerized Inspection System

SAMPLE NAME : DUKE 147
FILE NAME : DUKE147.072

DATE	: 12/08/1993	ACQ. RANGE	: 0.5-150	COUNTS	: 36964
TIME	: 09:23	ACQ. MODE	: SAMPLE	S.N.F.	: 0.74
CONFIG.	: 1 (0.7 31)	ACQ. TIME	: 189 SEC	S.D.U.	: 2895
CELL TYPE	: MAGNETIC (2)	SAMPLE SIZE	: 2	CONCENTR.	: 3.2E+05 #/ml
SAMPLE TYPE	: REGULAR	ACQ. CONF.	: 95.00 %(V)	SOLIDS	: 9.5E-03 %

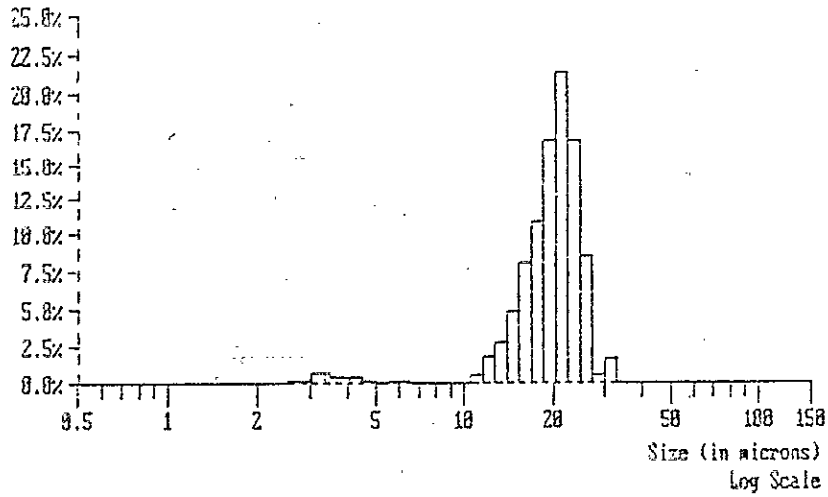
M+TE WC02018
Proc. ALO-530

PROBABILITY VOLUME DENSITY GRAPH

Name: DUKE 147
9.5E-05 cc/ml(100.0%)
Mode at 21.39 µm.
<< SCALE RANGE (µm): ADJUSTED >>

Mean(nv): 8.29µm
S.D.(nv): 6.14µm

Median : 20.32µm
Mean(vm): 19.57µm
S.D.(vm): 4.99µm
Conf(vm): 99.28 %



IA- 188

GALAI - CIS-1
Computerized Inspection System

SAMPLE NAME : 93-10374-M1/T-102 CORE 55
FILE NAME : 9310374.001

DATE	: 12/08/1993	ACQ. RANGE	: 0.5-150	COUNTS	: 23832
TIME	: 13:50	ACQ. MODE	: SAMPLE	S.N.F.	: 0.89
CONFIG.	: 1 (0.7 SI)	ACQ. TIME	: 232 SEC	S.D.U.	: 1132
CELL TYPE	: MAGNETIC (2)	SAMPLE SIZE	: 2	CONCENTR.	: 2.7E+05 #/ml
SAMPLE TYPE	: REGULAR	REQ. CONF.	: 95.00 %(V)	SOLIDS	: 1.4E-02 %

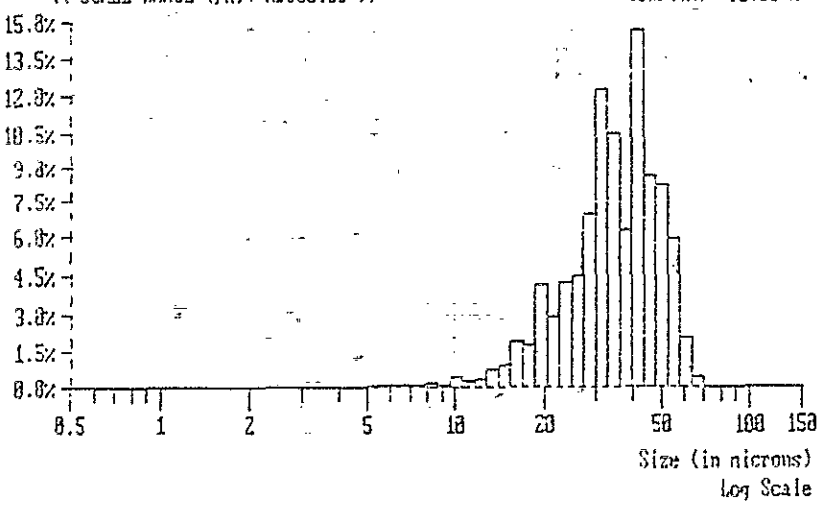
M+TE WO2018
Proc. ALO-530

PROBABILITY VOLUME DENSITY GRAPH

Name: 93-10374-M1/T-102 CORE 55
1.4E-04 cc/ml(100.0%)
Mode at 41.61 µm
<< SCALE RANGE (µm): ADJUSTED >>

Mean(nv): -0.90µm
S.D.(nv): 9.38µm

Median : 35.13µm
Mean(vm): 35.62µm
S.D.(vm): 11.94µm
Conf(vm): 95.00 %



IA-189

C05-005

VOLUME DISTRIBUTION TABLE (RANGED)

SAMPLE NAME : 93-10374-M1/T-102 CORE 55
FILE NAME : 9310374.001

DATE	: 12/08/1993	ACQ. RANGE	: 0.5-150	COUNTS	: 23802
TIME	: 13:50	ACQ. MODE	: SAMPLE	S.N.F.	: 0.59
CONFIG.	: 1 (0.7 SI)	ACQ. TIME	: 252 SEC	S.O.U.	: 1132
CELL TYPE	: MAGNETIC (2)	SAMPLE SIZE	: 2	CONCENTR.	: 2.7E+05 #/ml
SAMPLE TYPE	: REGULAR	REQ. CONF.	: 95.00 %(V)	SOLIDS	: 1.4E-02 %

RANGE (microns)	LOCAL (%)	UNDER(%)-CUMULATIVE-OVER(%)
0.0 - 1.0	0.02	0.02 99.98
1.0 - 2.0	0.08	0.10 99.90
2.0 - 3.0	0.05	0.15 99.85
3.0 - 4.0	0.07	0.23 99.77
4.0 - 5.0	0.15	0.38 99.62
5.0 - 6.0	0.13	0.51 99.49
6.0 - 7.0	0.16	0.67 99.33
7.0 - 8.0	0.17	0.84 99.16
8.0 - 9.0	0.23	1.07 98.93
9.0 - 10.0	0.25	1.32 98.68
10.0 - 20.0	8.56	10.88 89.12
20.0 - 30.0	20.76	31.65 68.15
30.0 - 40.0	28.38	60.73 39.27
40.0 - 50.0	25.42	86.15 13.85
50.0 - 60.0	12.53	98.67 1.33
60.0 - 70.0	1.33	100.00 0.00
70.0 - 80.0	0.00	100.00 0.00
80.0 - 90.0	0.00	100.00 0.00
90.0 - 100.0	0.00	100.00 0.00
100.0 - 150.0	0.00	100.00 0.00

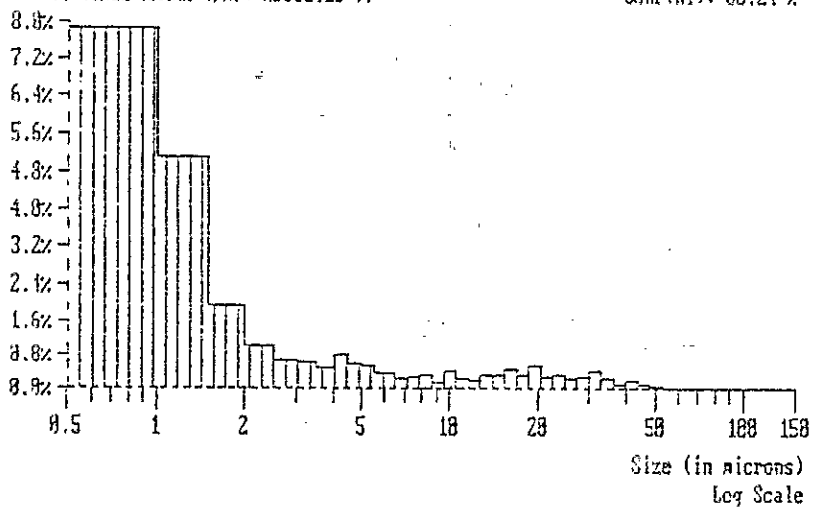
SAMPLE NAME : 93-10374-H1/T-102 CORE 55
FILE NAME : 9310374.001

DATE	: 12/09/1993	ACQ. RANGE	: 0.5-150	COUNTS	: 23832
TIME	: 13:50	ALG. MODE	: SAMPLE	S.N.F.	: 0.89
CONFIG.	: 1 (0.7 SI)	ACQ. TIME	: 232 SEC	S.D.U.	: 1132
CELL TYPE	: MAGNETIC (E)	SAMPLE SIZE	: E	CONCENTR.	: 2.7E+05 #/ml
SAMPLE TYPE	: REGULAR	REQ. CONF.	: 95.00 %(V)	SOLIDS	: 1.4E-02 %

PROBABILITY NUMBER DENSITY GRAPH

Name: 93-10374-H1/T-102 CORE 55
2.7E+05 #/ml(100.0%)
Mode at 8.75 µm
<< SCALE RANGE (µm): ADJUSTED >>

Median : 8.33µm
Mean(ml): 2.35µm
S.D.(ml): 5.31µm
Conf(ml): 80.24 %



NUMBER DISTRIBUTION TABLE (RANGES)

SAMPLE NAME : 93-10374-M1/T-108 CORE 55
FILE NAME : 9310374.001

DATE	: 12/08/1993	ACQ. RANGE	: 0.5-150	COUNTS	: 23832
TIME	: 13:50	ACQ. MODE	: SAMPLE	S.N.F.	: 0.99
CONFIG.	: 1 (0.7 "S1")	ACQ. TIME	: 232 SEC	S.D.U.	: 1132
CELL TYPE	: MAGNETIC (2)	SAMPLE SIZE	: 2	CONCENTR.	: 2.7E+05 #/ml
SAMPLE TYPE	: REGULAR	RCW. CONF.	: 95.00 %(V)	SOLIDS	: 1.4E-02 %

RANGE (microns)	LOCAL (%)	UNDER(%)-CUMULATIVE-OVER(%)	
0.0 - 1.0	57.52	57.52	42.48
1.0 - 2.0	27.83	85.35	14.65
2.0 - 3.0	3.71	89.06	10.94
3.0 - 4.0	1.76	90.82	9.18
4.0 - 5.0	1.69	92.51	7.49
5.0 - 6.0	0.80	93.31	6.69
6.0 - 7.0	0.61	93.92	6.08
7.0 - 8.0	0.41	94.33	5.67
8.0 - 9.0	0.39	94.72	5.28
9.0 - 10.0	0.35	94.99	5.01
10.0 - 20.0	2.51	97.50	2.50
20.0 - 30.0	1.36	98.86	1.14
30.0 - 40.0	0.75	99.61	0.39
40.0 - 50.0	0.30	99.91	0.09
50.0 - 60.0	0.08	99.99	0.01
60.0 - 70.0	0.01	100.00	0.00
70.0 - 80.0	0.00	100.00	0.00
80.0 - 90.0	0.00	100.00	0.00
90.0 - 100.0	0.00	100.00	0.00
100.0 - 150.0	0.00	100.00	0.00

IA-192

C05-008

G A L A I — C I S — 1
Computerized Inspection System

SAMPLE NAME : 93-10374-M2/T-102 CORE 55
FILE NAME : 9310374.002

DATE	: 12/08/1993	ACQ. RANGE	: 0.5-150	COUNTS	: 37982
TIME	: 14:07	ACQ. MODE	: SAMPLE	S.N.F.	: 0.50
CONFIG.	: 1 (0.7 SI)	ACQ. TIME	: 262 SEC	S.D.U.	: 2034
CELL TYPE	: MAGNETIC (2)	SAMPLE SIZE	: 2	CONCENTR.	: 2.5E+05 #/ml
SAMPLE TYPE	: REGULAR	REQ. CONF.	: 95.00 %(V)	SOLIDS	: 1.7E-02 %

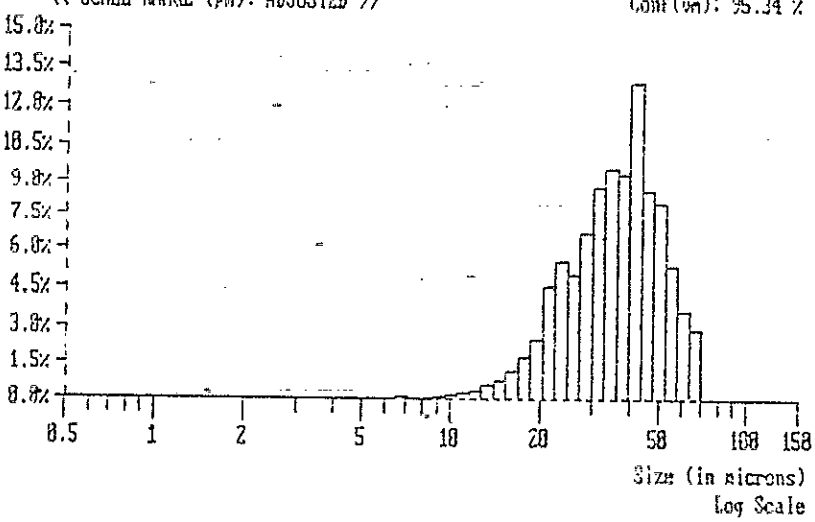
M+TE WC02018
Proc. ALO-530

PROBABILITY VOLUME DENSITY GRAPH

Name: 93-10374-M2/T-102 CORE 55
1.7E-04 cc/ml(100.0%)
Mode at 41.61 μm
<< SCALE RANGE (μm): ADJUSTED >>

Mean(nv): 10.81μm
S.D.(nv): 10.12μm

Median : 36.57μm
Mean(vm): 36.92μm
S.D.(vm): 12.78μm
Conf(vm): 95.34 %



14-193

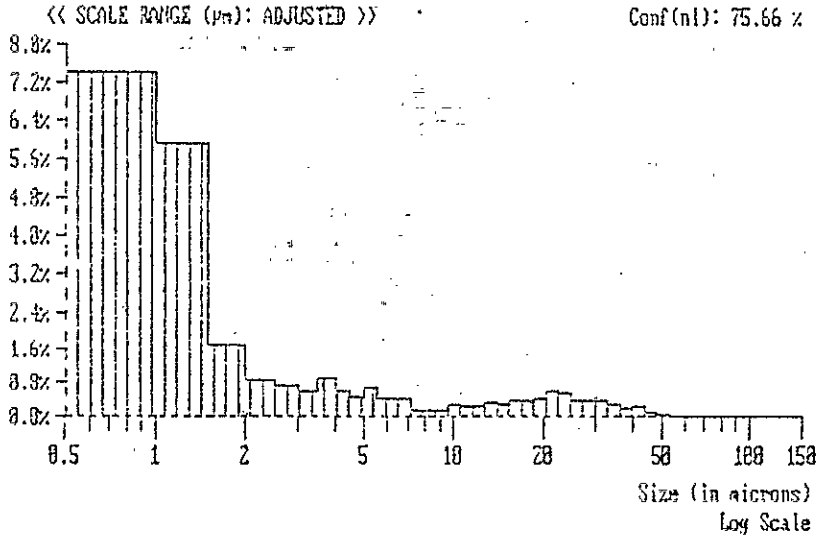
SAMPLE NAME : 93-10374-M2/T-102 CORE 55
FILE NAME : 9310374.002

DATE	: 12/08/1993	ACQ. RANGE	: 0.5-150	COUNTS	: 37962		
TIME	: 14:07	ACQ. MODE	: SAMPLE	S.N.F.	: 0.50		
CONFIG.	: 1 (0.7 61)	ACQ. TIME	: 262 SEC	S.D.U.	: 2034		
CELL TYPE	: MAGNETIC (2)	SAMPLE SIZE	: 2	CONCENTR.	: 2.5E+05	#/ml	
SAMPLE TYPE	: REGULAR	REQ. CONF.	: 95.00 %(V)	SOLIDS	: 1.7E-02	%	

PROBABILITY NUMBER DENSITY GRAPH

Name: 93-10374-M2/T-102 CORE 55
2.5E+05 #/ml(100.0%)
Mode at 0.75 µm
<< SCALE RANGE (µm): ADJUSTED >>

Median : 0.96µm
Mean(nl): 2.57µm
S.D.(nl): 5.87µm
Conf(nl): 75.66 %



/A-194

C05-010

VOLUME DISTRIBUTION TABLE (RANGES)

SAMPLE NAME : 93-10374-M2/T-108 CORE 55
FILE NAME : 9310374.002

DATE	: 12/08/1993	ACQ. RANGE	: 0.3-150	COUNTS	: 37982
TIME	: 14:07	ACQ. MODE	: SAMPLE	S.N.F.	: 0.50
CONFIG.	: 1 (0.7 SI)	ACQ. TIME	: 262 SEC	S,D.U.	: 2034
CELL TYPE	: MAGNETIC (2)	SAMPLE SIZE	: 2	CONCENTR.	: 2.5E+05 #/ml
SAMPLE TYPE	: REGULAR	REQ. CONF.	: 95.00 %(V)	SOLIDS	: 1.7E-02 %

RANGE (microns)	LOCAL (%)	UNDER(%)-CUMULATIVE-OVER(%)	
0.0 - 1.0	0.02	0.02	99.98
1.0 - 2.0	0.06	0.08	99.92
2.0 - 3.0	0.04	0.12	99.88
3.0 - 4.0	0.08	0.20	99.80
4.0 - 5.0	0.09	0.29	99.71
5.0 - 6.0	0.10	0.39	99.61
6.0 - 7.0	0.20	0.59	99.41
7.0 - 8.0	0.07	0.66	99.34
8.0 - 9.0	0.10	0.75	99.25
9.0 - 10.0	0.17	0.92	99.08
10.0 - 20.0	6.99	7.91	92.09
20.0 - 30.0	23.04	30.95	69.05
30.0 - 40.0	28.09	59.04	40.96
40.0 - 50.0	23.54	82.59	17.41
50.0 - 60.0	14.43	97.02	2.98
60.0 - 70.0	2.98	100.00	0.00
70.0 - 80.0	0.00	100.00	0.00
80.0 - 90.0	0.00	100.00	0.00
90.0 - 100.0	0.00	100.00	0.00
100.0 - 150.0	0.00	100.00	0.00

NUMBER DISTRIBUTION TABLE (RANGES)

SAMPLE NAME : 93-10374-M2/T-102 CORE 55
FILE NAME : 9310374.002

DATE	: 12/08/1993	ACQ. RANGE	: 0.3-150	COUNTS	: 37982
TIME	: 14:07	ACQ. MODE	: SAMPLE	S.N.F.	: 0.50
CONFIG.	: 1 (0.7 31)	ACQ. TIME	: 252 SEC	S.D.U.	: 2034
CELL TYPE	: MAGNETIC (2)	SAMPLE SIZE	: 2	CONCENTR.	: 2.5E+05 #/ml
SAMPLE TYPE	: REGULAR	REQ. CONF.	: 95.00 %(V)	SOLIDS	: 1.7E-02 %

RANGE (microns)	LOCAL (%)	UNDER(%)-CUMULATIVE-OVER(%)	
0.0 - 1.0	54.24	54.24	45.76
1.0 - 2.0	30.55	84.79	15.21
2.0 - 3.0	3.48	88.27	11.73
3.0 - 4.0	2.28	90.55	9.45
4.0 - 5.0	1.22	91.76	8.24
5.0 - 6.0	0.84	92.60	7.40
6.0 - 7.0	0.94	93.54	6.46
7.0 - 8.0	0.21	93.75	6.25
8.0 - 9.0	0.20	93.95	6.05
9.0 - 10.0	0.24	94.19	5.81
10.0 - 20.0	2.43	96.62	3.38
20.0 - 30.0	2.01	98.64	1.36
30.0 - 40.0	0.88	99.51	0.49
40.0 - 50.0	0.36	99.87	0.13
50.0 - 60.0	0.11	99.99	0.01
60.0 - 70.0	0.01	100.00	0.00
70.0 - 80.0	0.00	100.00	0.00
80.0 - 90.0	0.00	100.00	0.00
90.0 - 100.0	0.00	100.00	0.00
100.0 - 150.0	0.00	100.00	0.00

WHC-SD-WM-DP-052-
ADDENDUM 1 REV. 0

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

WT% SOLIDS DATA SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

CLIENT: TWC WORK PACKAGE: M99569 ASR/ARF/LOI/TI: TI-TWC-06

QA PLAN: ALO-003 IMPACT LEVEL: II PROCEDURE NUMBER: PNL-ALO-504

TANK T-102, CORE 55 SLUDGE
SAMPLE IDENTIFICATION

ACL NUMBER	Spl. CLIENT IDENTIFICATION	TARE WEIGHT (G)	(A) SAMPLE WET WEIGHT PLUS TARE	(B) SAMPLE DRY WEIGHT PLUS TARE	WEIGHT % SOLIDS
93-08755-K-1	C55-FIL	8.2097	8.7482	8.7434	99.11
93-08755-K-2	C55-FIL	8.2226	8.7253	8.7209	99.12

WT% SOLIDS = $\frac{B - TARE}{A - TARE} \times 100$

DATE/TIME IN: 8/9/93 1130 OVEN TEMPERATURE: 107 °C

DATE/TIME OUT: 8/10/93 0130 OVEN TEMPERATURE: 106 °C

BALANCE: CELL 2 (360-06-01-016) X

THERMOCOUPLE: ~~0186~~ SN 6311 RTS 8/13/93

BALANCE: CELL 6 (362-06-01-036) ___

Analyst: D Vaughn Rogers Date: 8/9/93 Reviewer: Ficke L Steele Date: 8/9/93

C06-003 11-297

0001185

DATE TO QC: September 2, 1993

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - pH (of Water Leach & Blanks)

Data Package/Report - Core 55

Project No. - 20777

ACL Numbers -				
		93-08755-M1	93-08755-M2	93-08755-M3
	Field Blank:	93-05874-P1	93-05874-P2	
	Hot-Cell Blank:	93-09774-P1	93-09774-P2	
	DIW:	93-09804-P1	93-09804-P2	

Thomas A Walker
PNL ACL Quality Representative

9/7/93
Date

14-198

C07-002

000186

Battelle
Pacific Northwest Laboratories

Project Number _____

Internal Distribution

Date September 1, 1993

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

To SG McKinley

From MC Burt

Subject pH Measurement of Water Leach and Blanks from T-102

The water leach solutions from T-102, Core 55 and the various related blank solutions were measured for solution pH using procedure ALO-225. M&TE used was Titroprocessor 672, WB76896. Data is recorded in T-102 Data File in Rm.104 and in the ALO Office. The electrode was calibrated with current NIST traceabale pH buffer solutions at pH 4 and 10 and calibration checked at pH 7.

A deviation from the test plan was required because pH could not be measured directly on the solid sample and there was insufficient sample to perform a 1:1 dilution. These measurements were performed on a 1:5 dilution.

ALO Number	Sample Ident.	Obs. pH
93-05874-P1	T-102 Field Blank	8.47
93-05874-P2	"	8.48
93-09774-P1	T-102 Hot Cell Blank	8.44
93-09774-P2	"	8.48
93-09804-P1	T-102 HLRF DIW	7.94
93-09804-P2	"	7.93
93-08755-M1	T-102 Core 55	9.80
93-08755-M2	"	9.83
93-08755-M3	T-102 Blank	7.49

If there are any questions regarding this result please contact me on 376-3762.

MC Burt, Sr. Res.Scientist
Analytical Chemistry Laboratory

Concur _____

C07-003 IA-199

000187

BATTELLE - PNL

ALO-225

pH

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

Date: 8/31/93 Analyst: MC Burt

Reviewer: DA Baldwin

M+TE WB76896

Calibration check points: 4 / 7 / 10

Buffer lot number: VWR 32111 / VWR 116 / BECKMAN S110151

Buffer exp. date: 4/94 / 10/94 / 10/93

Sample number	ALO number	Obs. pH
	pH 7 Buffer	7.04
FIELD BLANK	93-05874-P-1	8.47
"	93-05874-P-2	8.48
HLRF HotCell BLANK	93-09774-P1	8.44
"	93-09774-P-2	8.43
HLRF DIW	93-09804-P1	7.94
"	93-09804-P-2	7.93
T-102 CORE 55	93-08755-M-1	9.80
"	93-08755-M-2	9.83
BLANK	93-08755-M-3	7.49
	pH 7 Buffer	7.08

C07-004)A-200

000188

WHC-SD-WM-DP-052, ADDENDUM 1 REV. 0

BOHLIN CS RHEOMETER
Constant rate test
1993-08-10   18:46:44

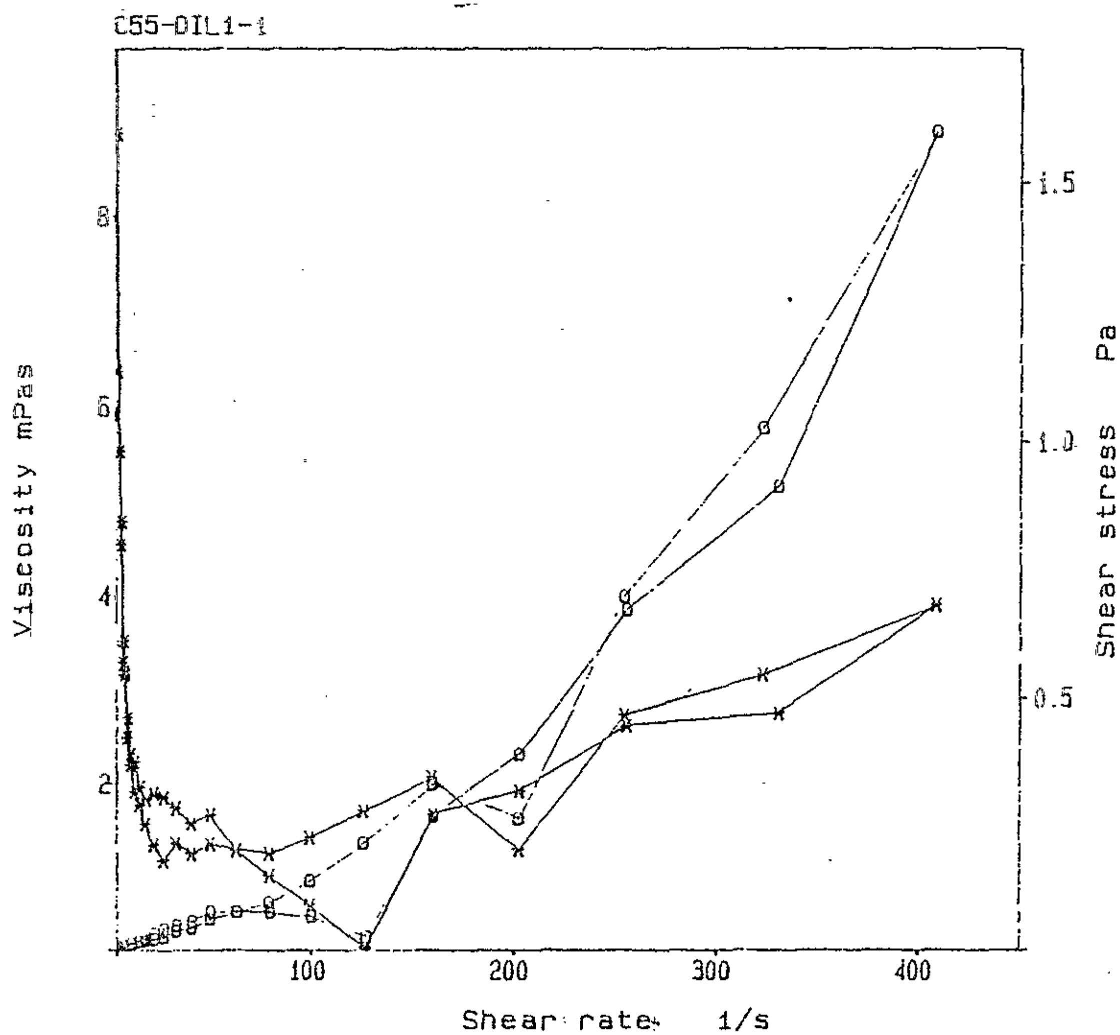
K-K Viscosity
— Shear stress

C 25

CDt 1 s   It 30 s
No of M 1
MI 10 s

T 24.9 - 25.1 C

C:\DATA\C55O11



C08-029

1A- 201

G00189

BOHLIN CS RHEOMETER
Constant rate test
1993-08-10   20:00:53

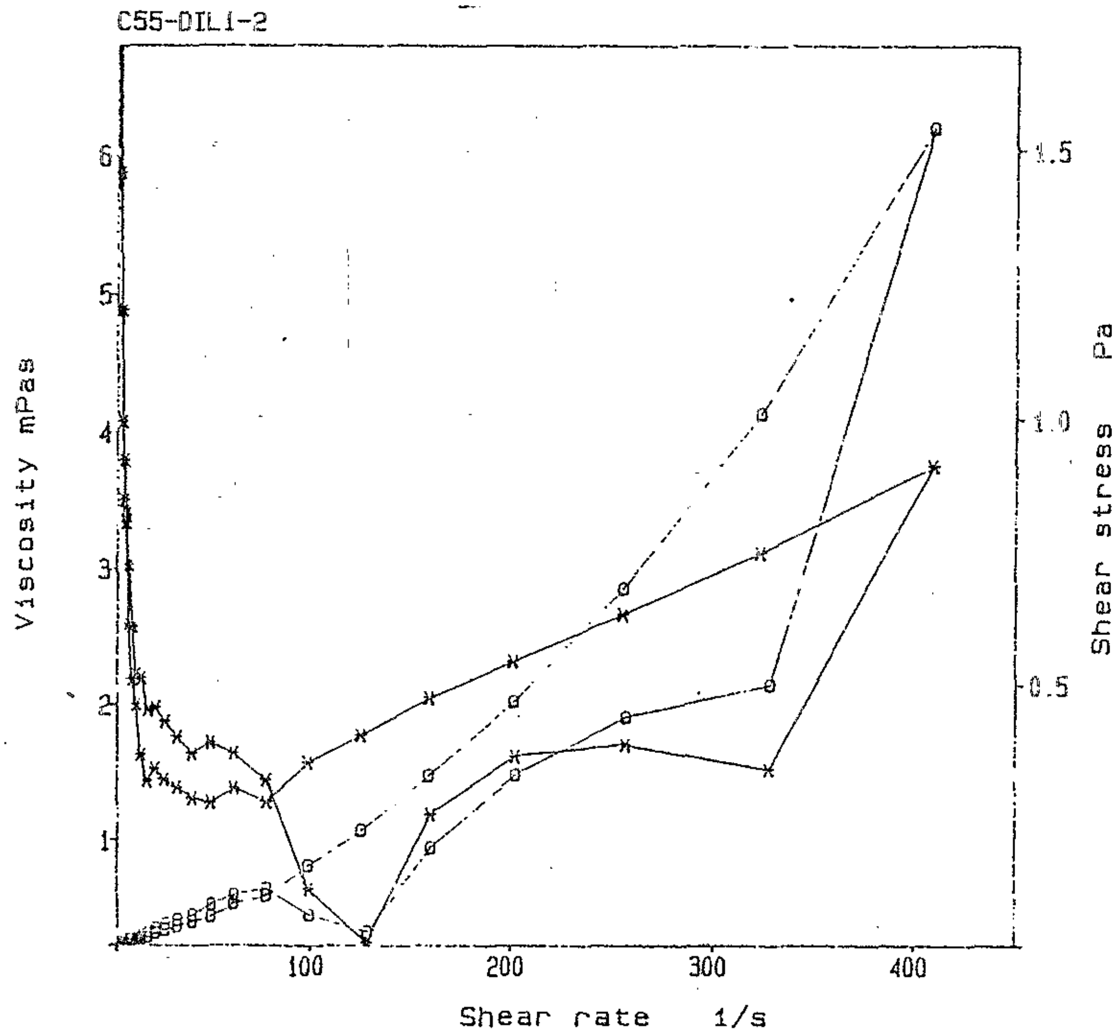
— Viscosity
— Shear stress

C 25

CDt 1 s   It 30 s
No of M 1
MI 10 s

T 24.9 - 25.1 C

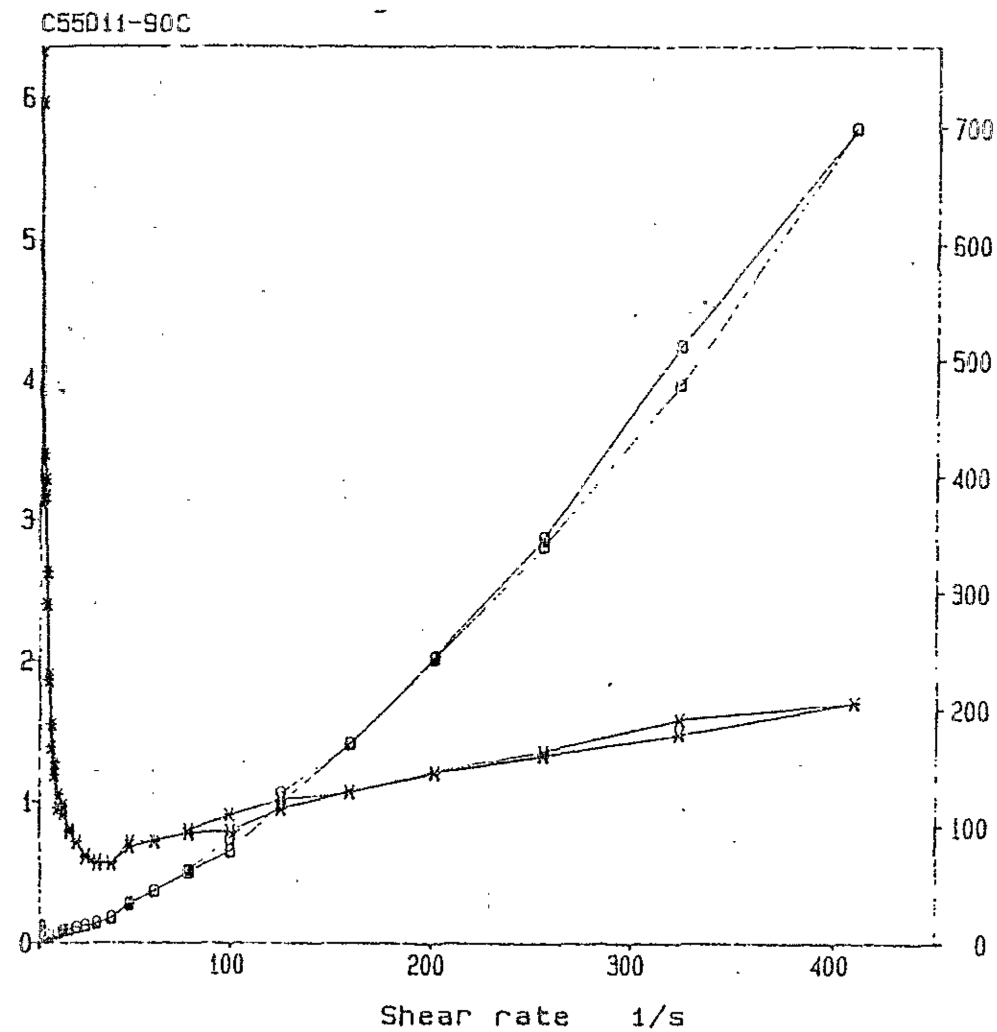
C:\DATA\C55D12



WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

/4-202
C08-020
C00190

BOHLIN CS RHEOMETER
Constant rate test
1993-08-11 17:02:14



C 25

COt 5 s It 30 s
No of M 1
MI 2 s

T 89.9 - 90.2 C

C:\DATA\C55D1190

/A-203

C08-031

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

BOHLIN CS RHEOMETER
Constant rate test
1993-08-11 17:40:48

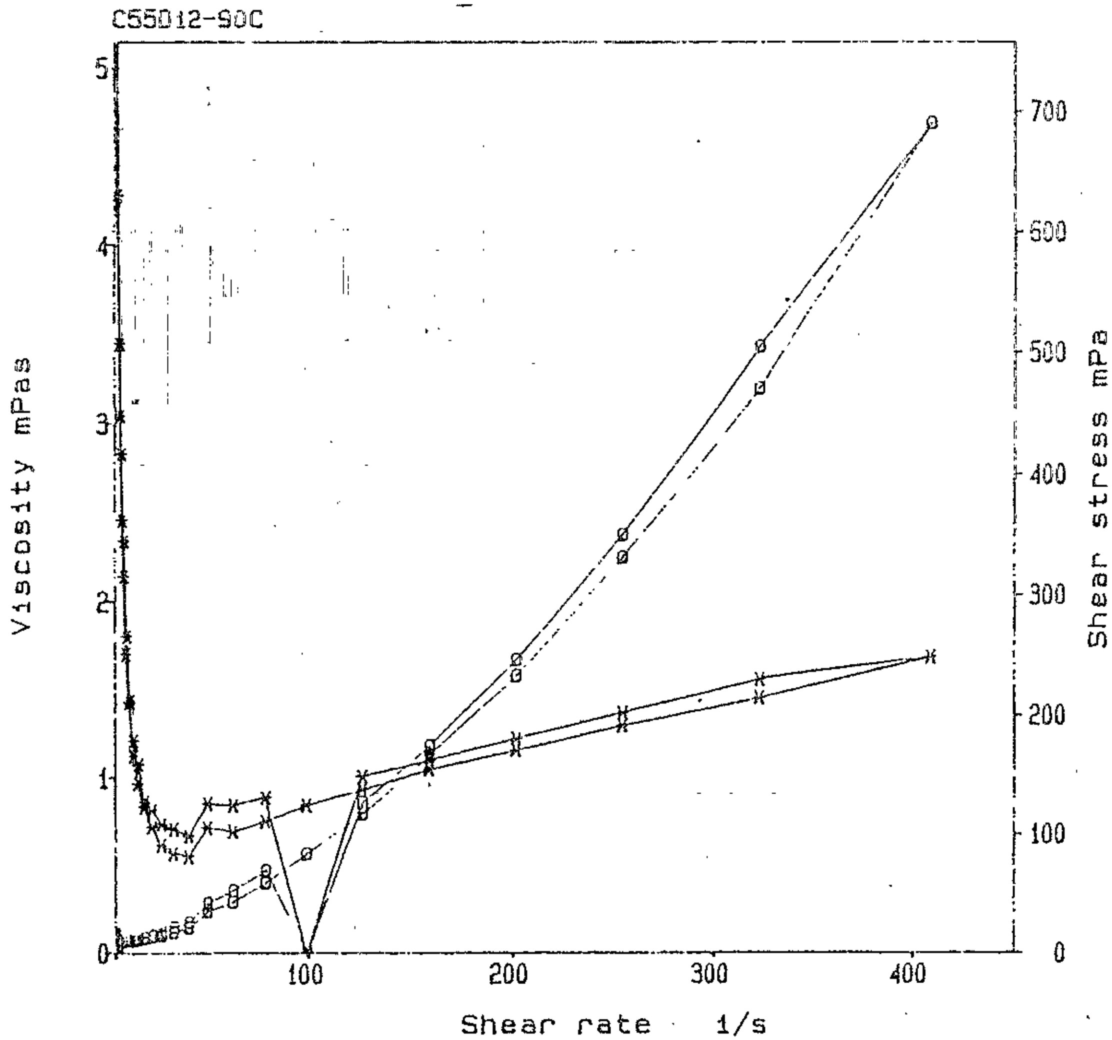
— Viscosity
— Shear stress

C 25

COt 5 s It 30 s
No of M 1
MI 2 s

T 89.9 - 90.1 C

C:\DATA\C55D1290



14-201
C08-032
C0G192

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

BOHLIN CS RHEOMETER
Constant rate test
1993-08-30 23:44:44

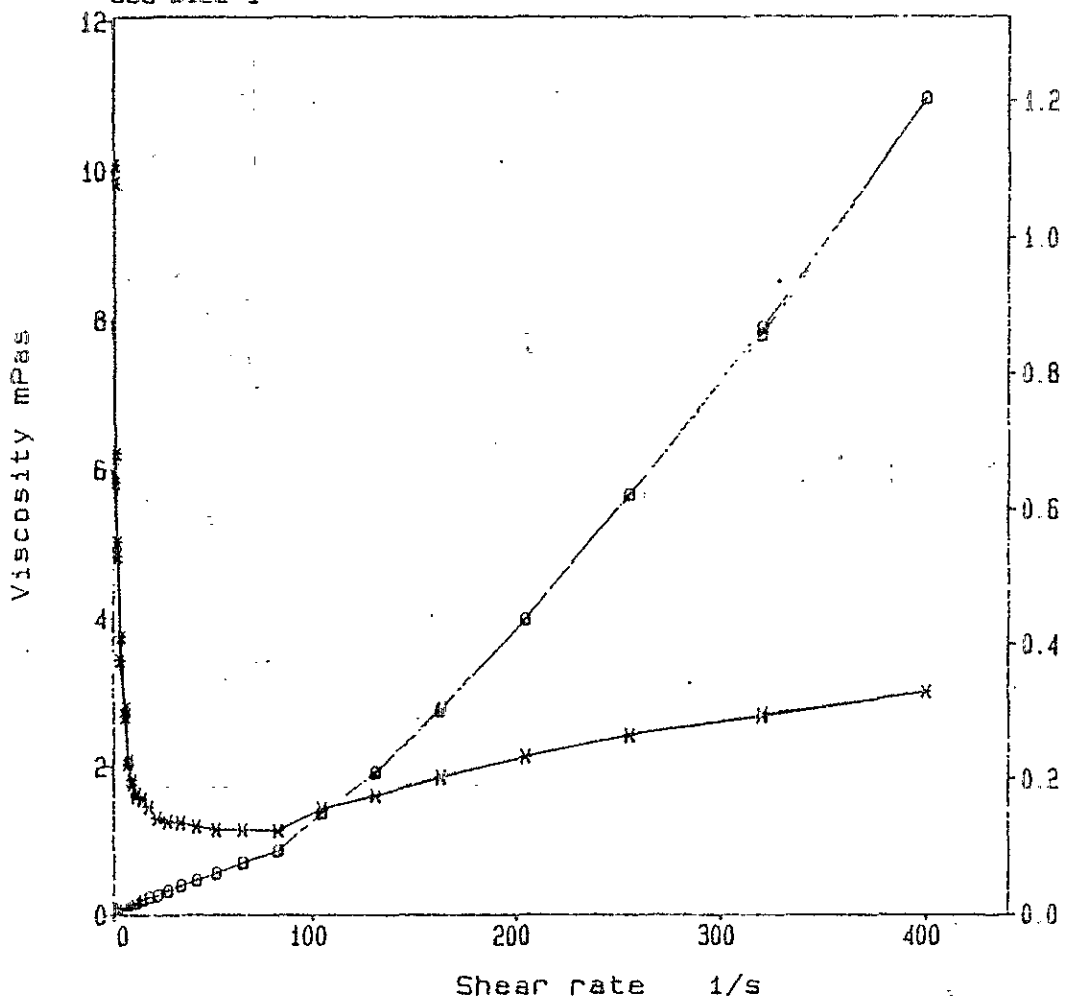
— Viscosity
— Shear stress

C 25

Cdt 10 s It 30 s.
No of M 1
MI 2 s

T 24.9 - 25.1 C

C55 DIL2-1



WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

/A-205
C08-033

0001193

BOHLIN CS RHEOMETER
Constant rate test
1993-08-31 00:52:21

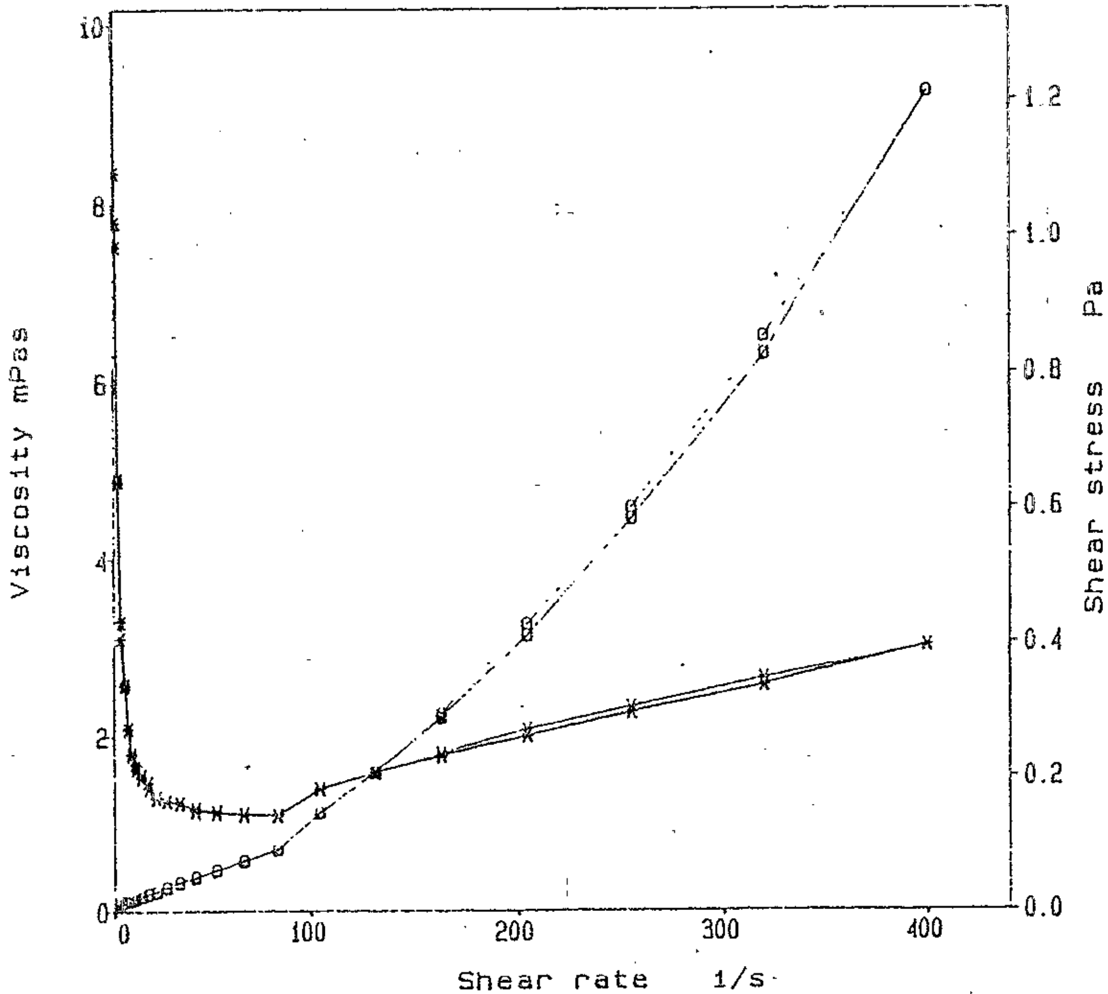
— Viscosity
— Shear stress

C 25

COt 10 s It 30 s
No of M 1
MI 2 s

T 24.9 - 25.2 C

C:\DATA\C55025



C08-034

H-206

C09194

BOHLIN CS RHEOMETER
Constant rate test
1998-08-31 02:48:51

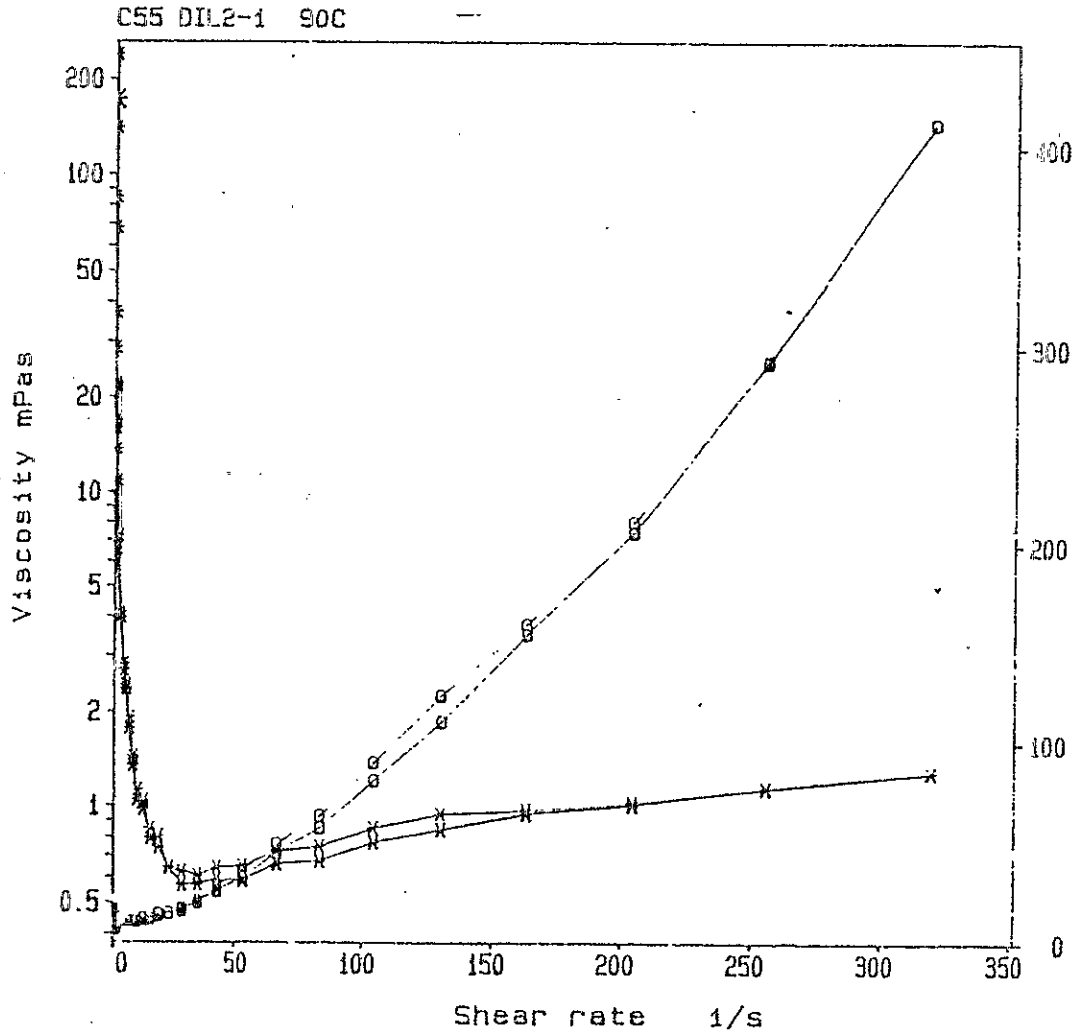
X-X Viscosity
0-0 Shear stress

C 25

CDt 10 s It 30 s
No of M 1
MI 2 s

T 89.9 - 90.1 C

C:\DATA\C55D2690



WHC-SD-WM-DP-052,
ADDENDUM 1 REV. 0

C08-035

/A-207

000195

BOHLIN CS RHEOMETER
Constant rate test
1993-08-31 09:30:38

Viscosity
Shear stress

d 25

Dt 10 s It 30 s
No of M 1
MI 2 s

T 89.9 - 90.2 C

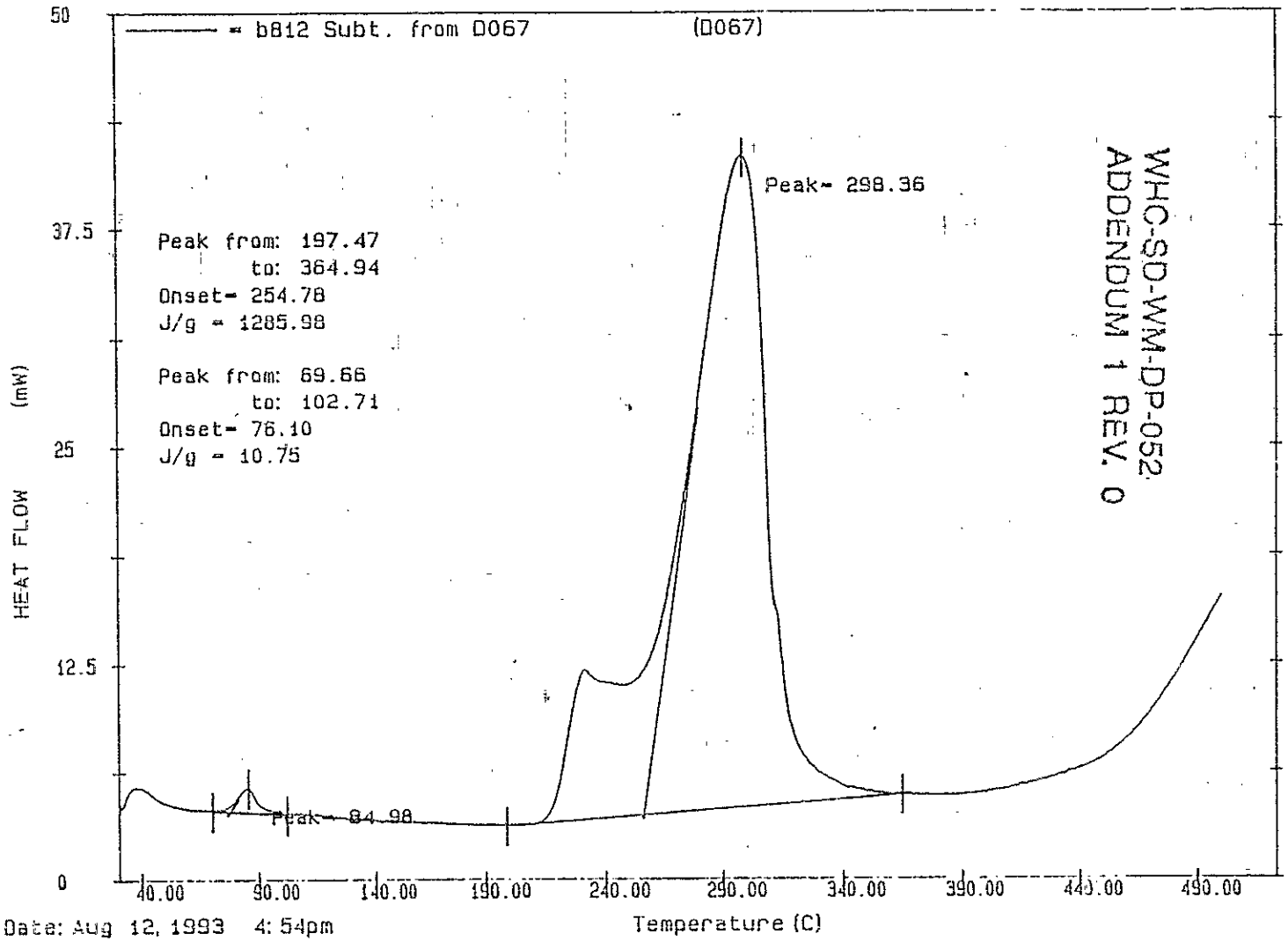
C:\DATA\C5502790

C55 DIL2-2 90C

Viscosity mPas

Shear stress mPa

Shear rate 1/s



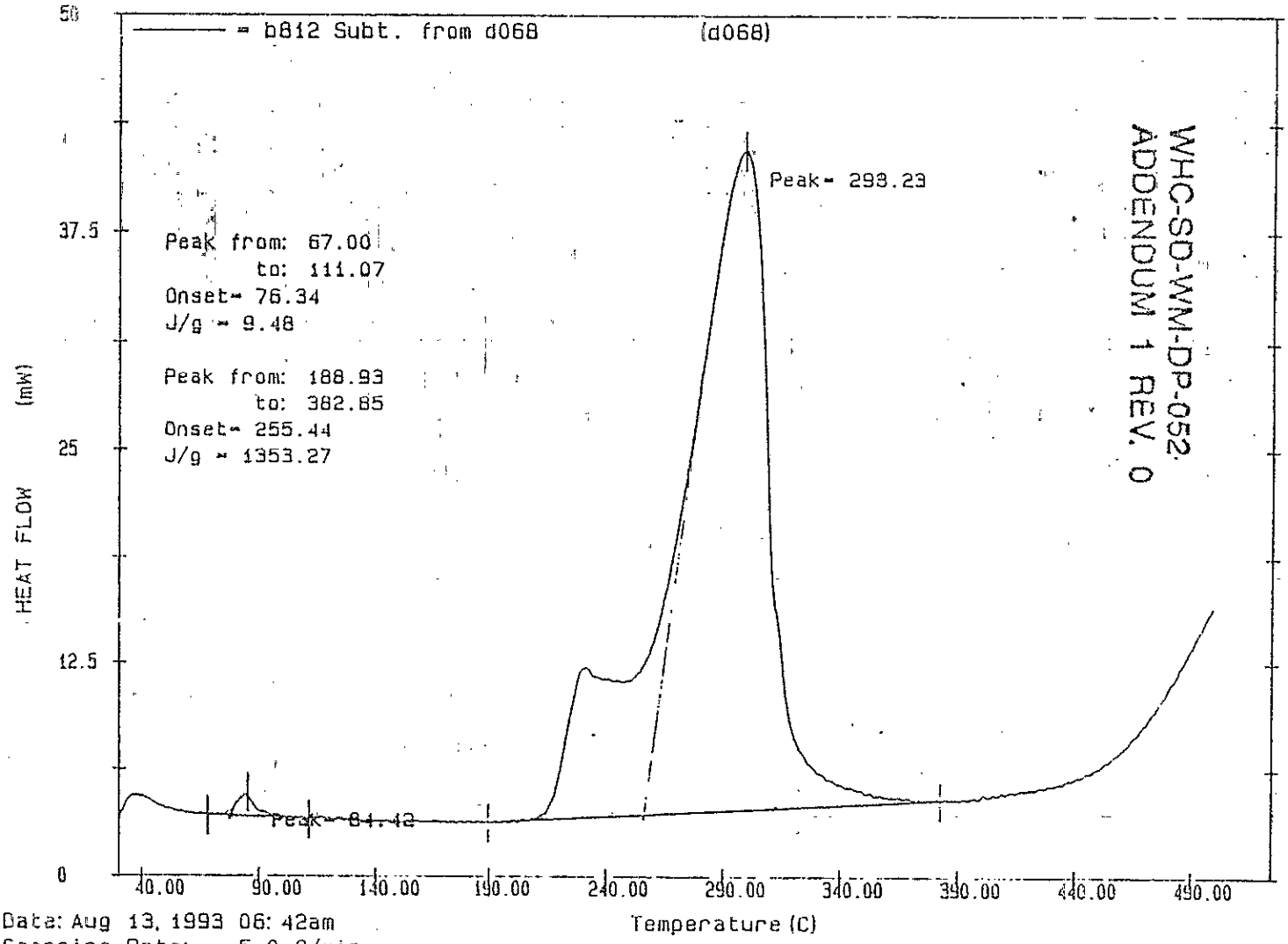
Date: Aug 12, 1993 4:54pm
Scanning Rate: 5.0 C/min
Sample Wt: 15.910 mg Path: a:\
File 1: D067 RLS

PERKIN-ELMER DSC7

C09-002

14-209

D0067



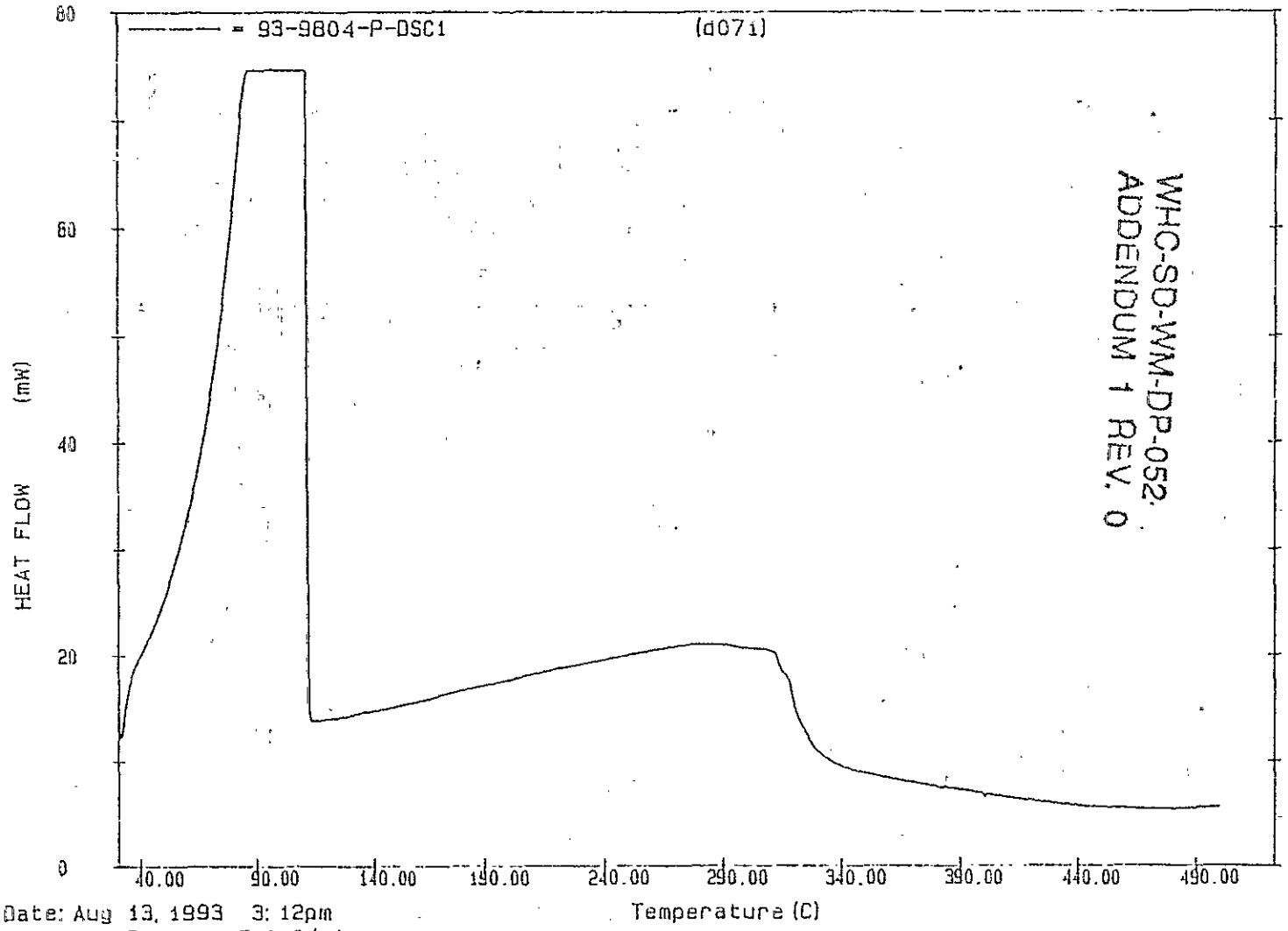
Data: Aug 13, 1993 06:42am
Scanning Rate: 5.0 C/min
Sample Wt: 15.730 mg Path: a:\
File 1: D068 RLS

PERKIN-ELMER DSC7

/ 4-210

C09-003

0001198



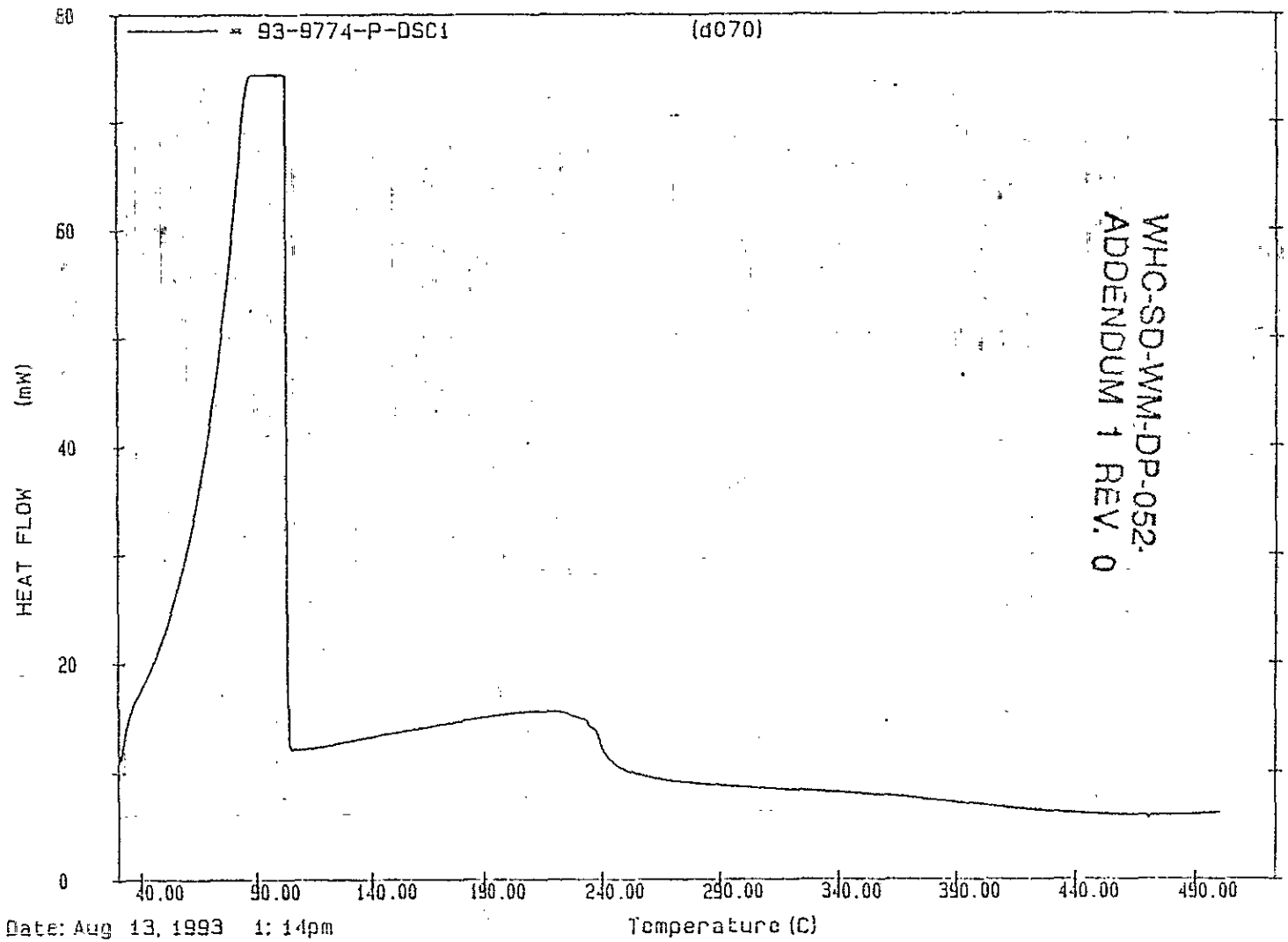
Date: Aug 13, 1993 3:12pm
Scanning Rate: 5.0 C/min
Sample Wt: 20.770 mg Path: a:\
File 1:0071 RLS

PERKIN-ELMER DSC7

H-21-211

C09-004

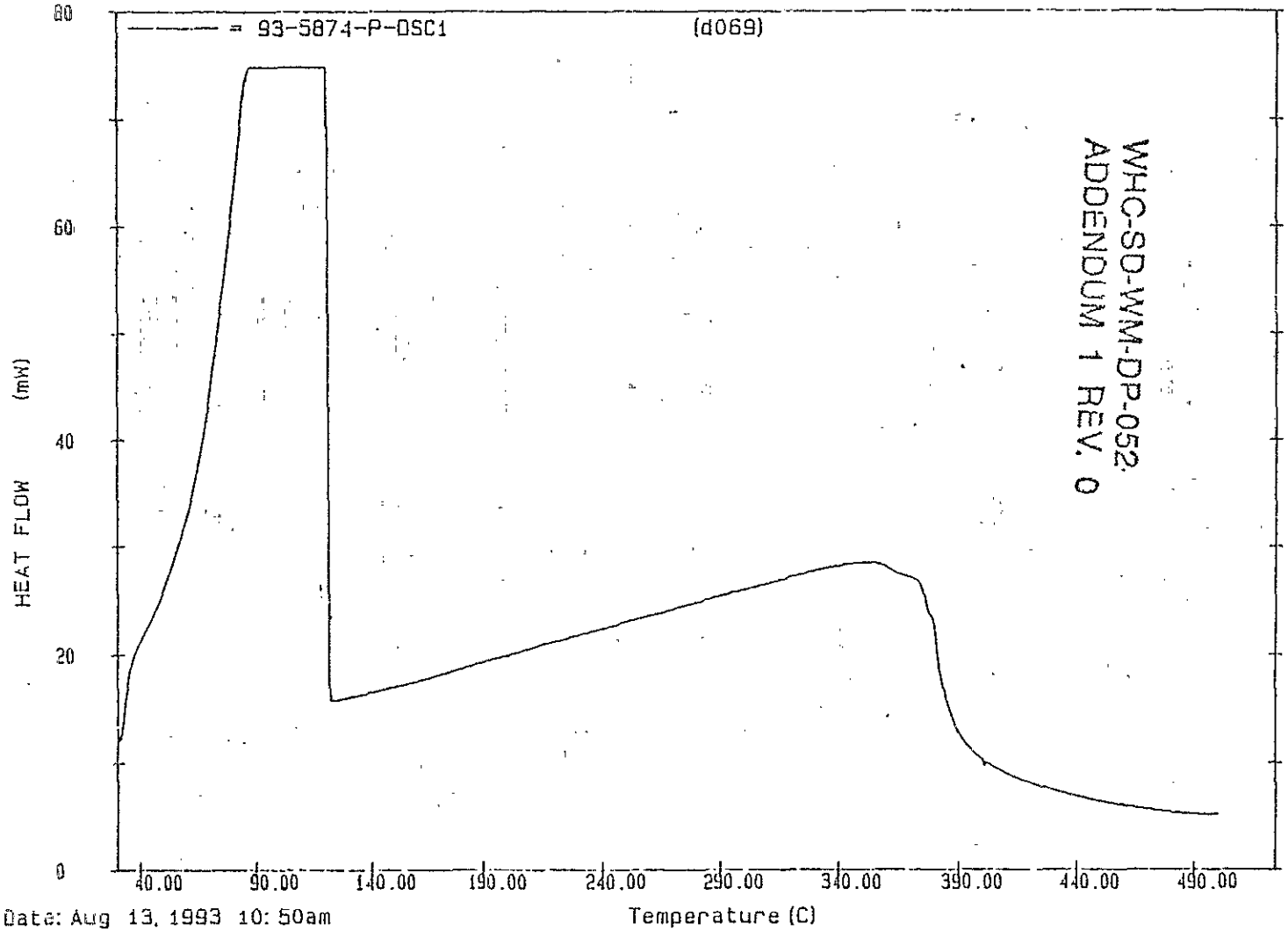
C00139



C09-005

2T22-#1

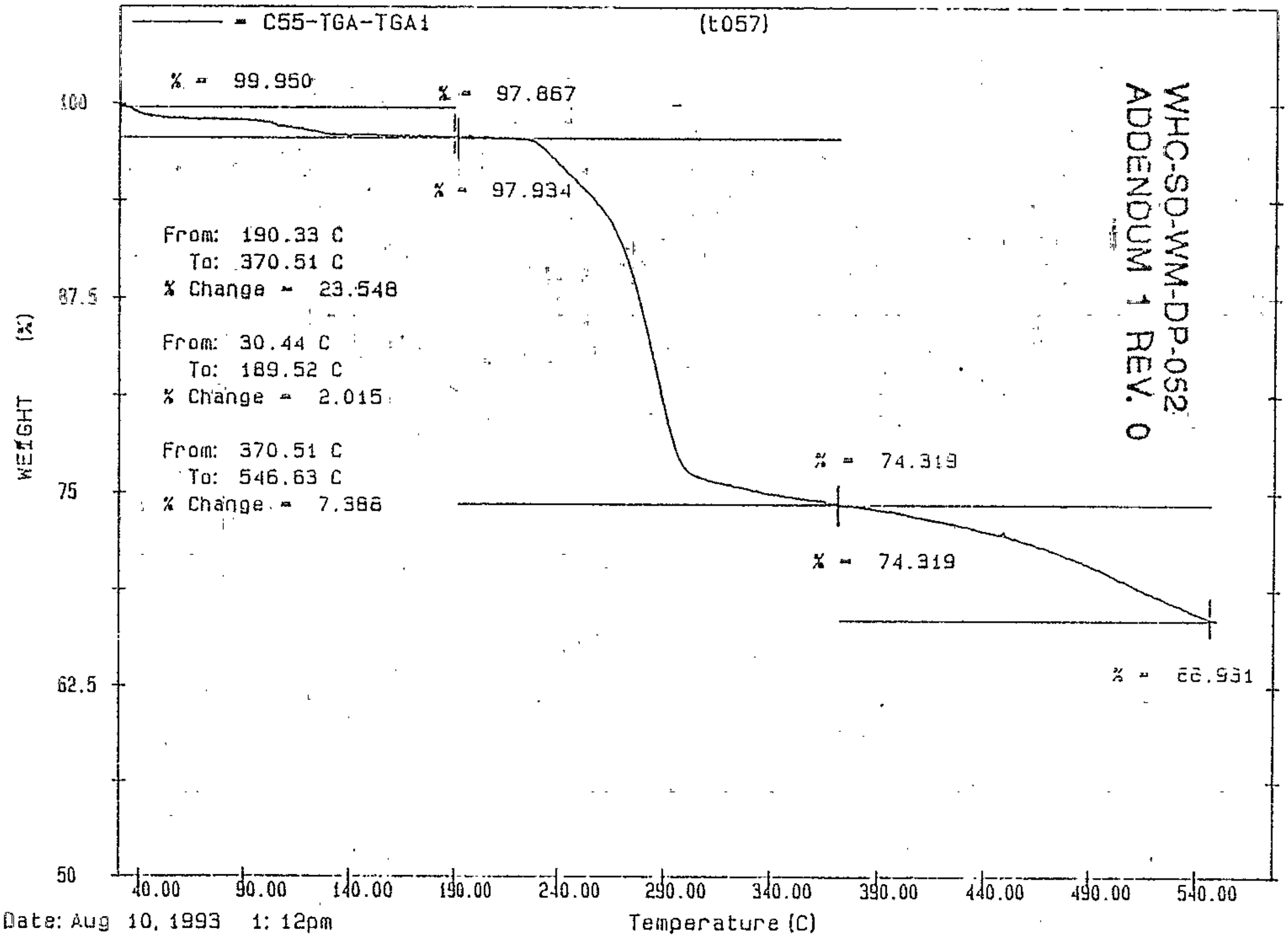
000200



WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

H-213
C09-006

D00201



WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

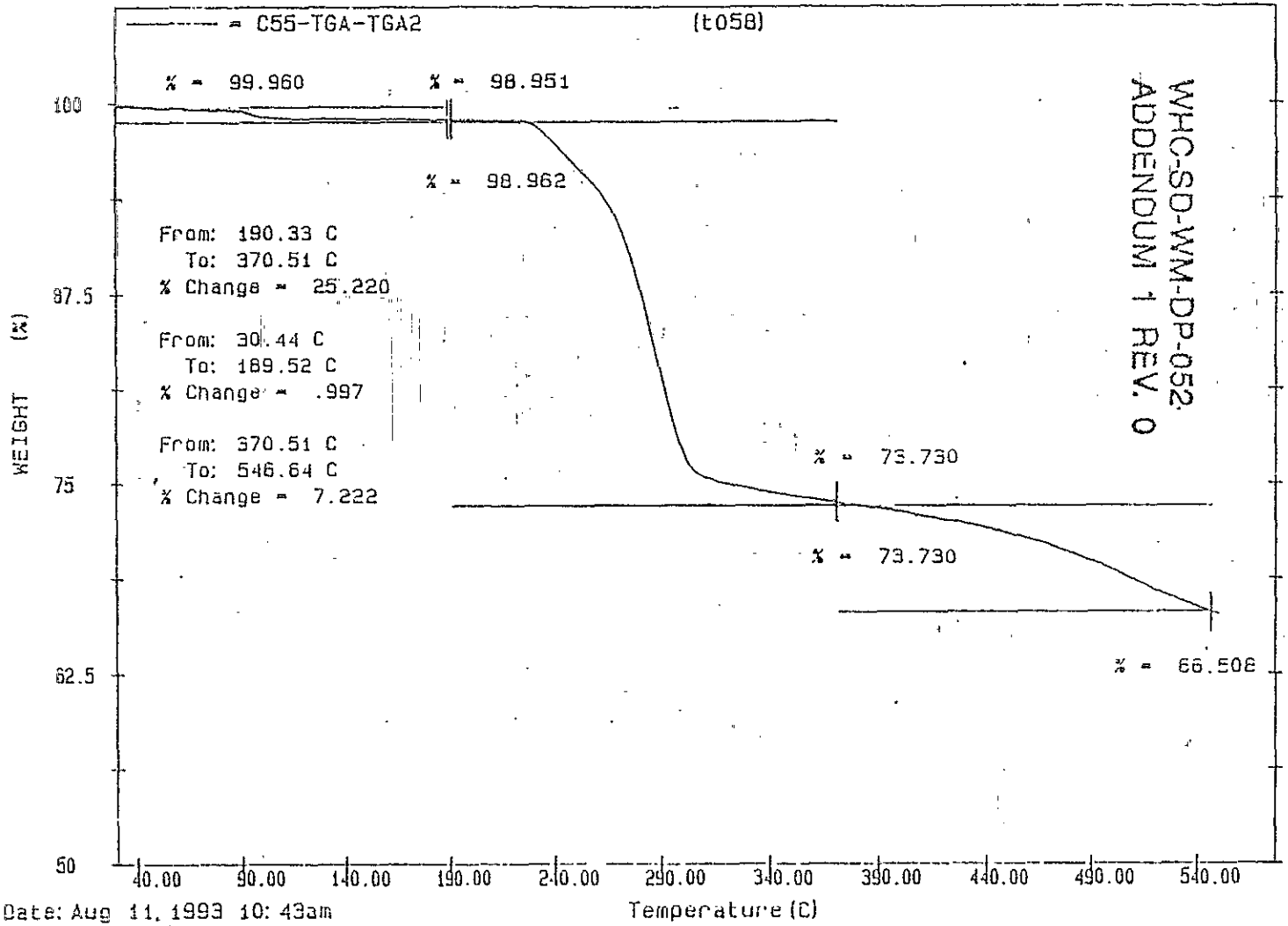
Date: Aug 10, 1993 1: 12pm
Scanning Rate: 5.0 C/min
Sample Wt: 6.276 mg Path: a: \
File: T057 *RLS*

Perkin-Elmer TGA7

C09-007

A-214

0002020

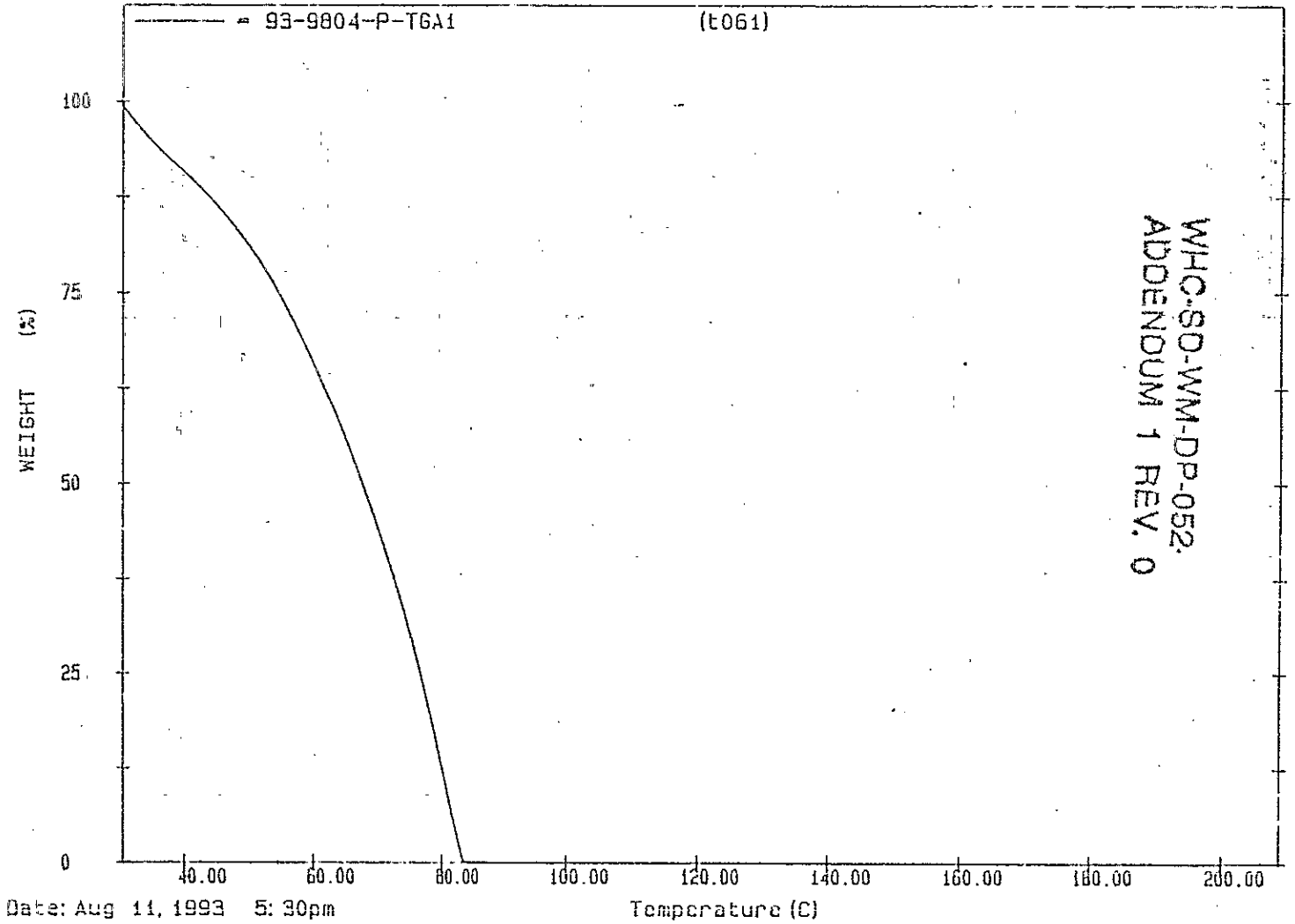


Date: Aug 11, 1993 10:43am
Scanning Rate: 5.0 C/min
Sample Wt: 7.076 mg Path: a:\
File: T058 RLS

Perkin-Elmer TGA7

1H-215
C09-008

000203



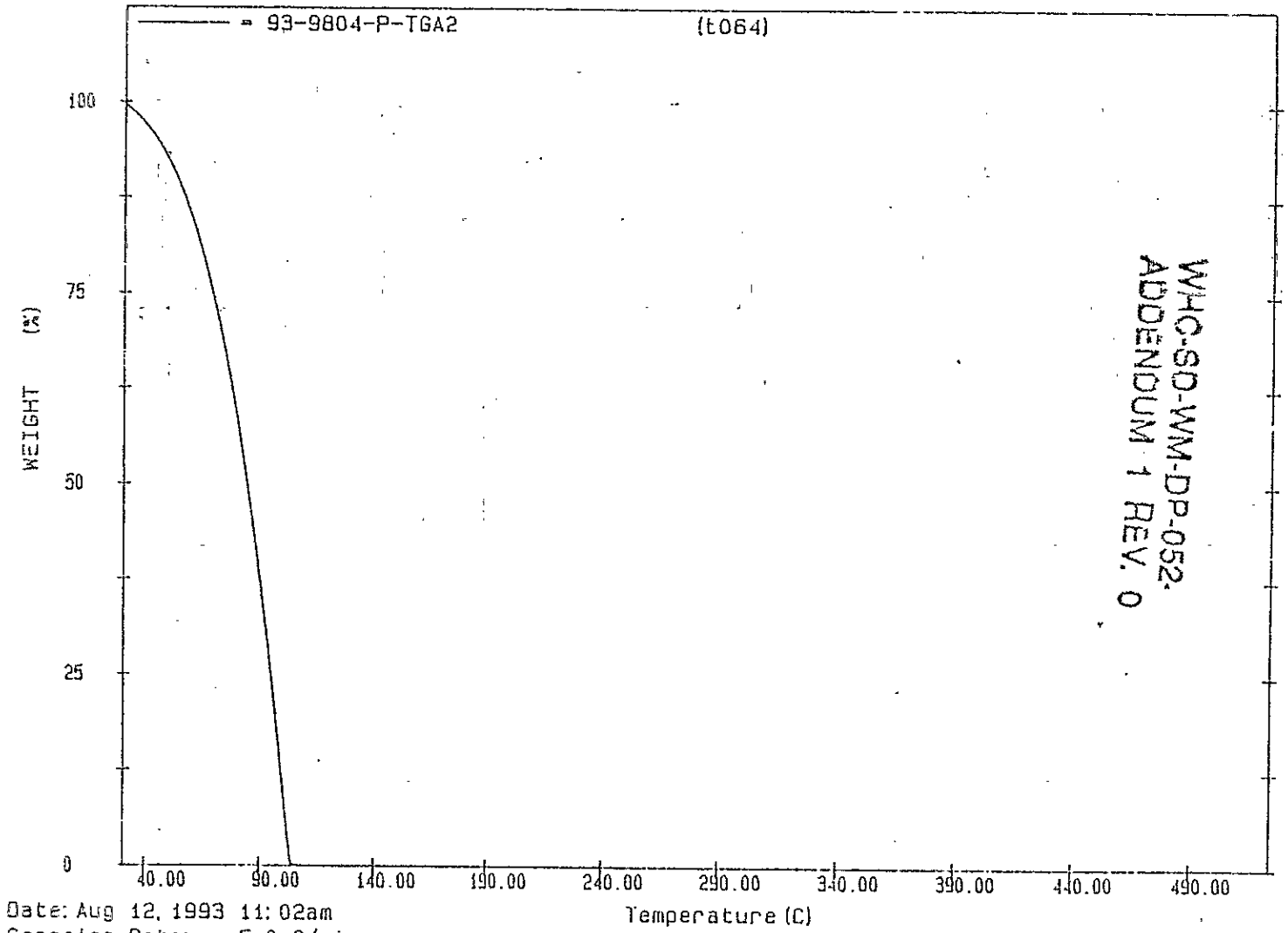
WHC-SD-WM-DP-052,
ADDENDUM 1 REV. 0

Perkin-Elmer TGA7

14-216

C09-009

403003



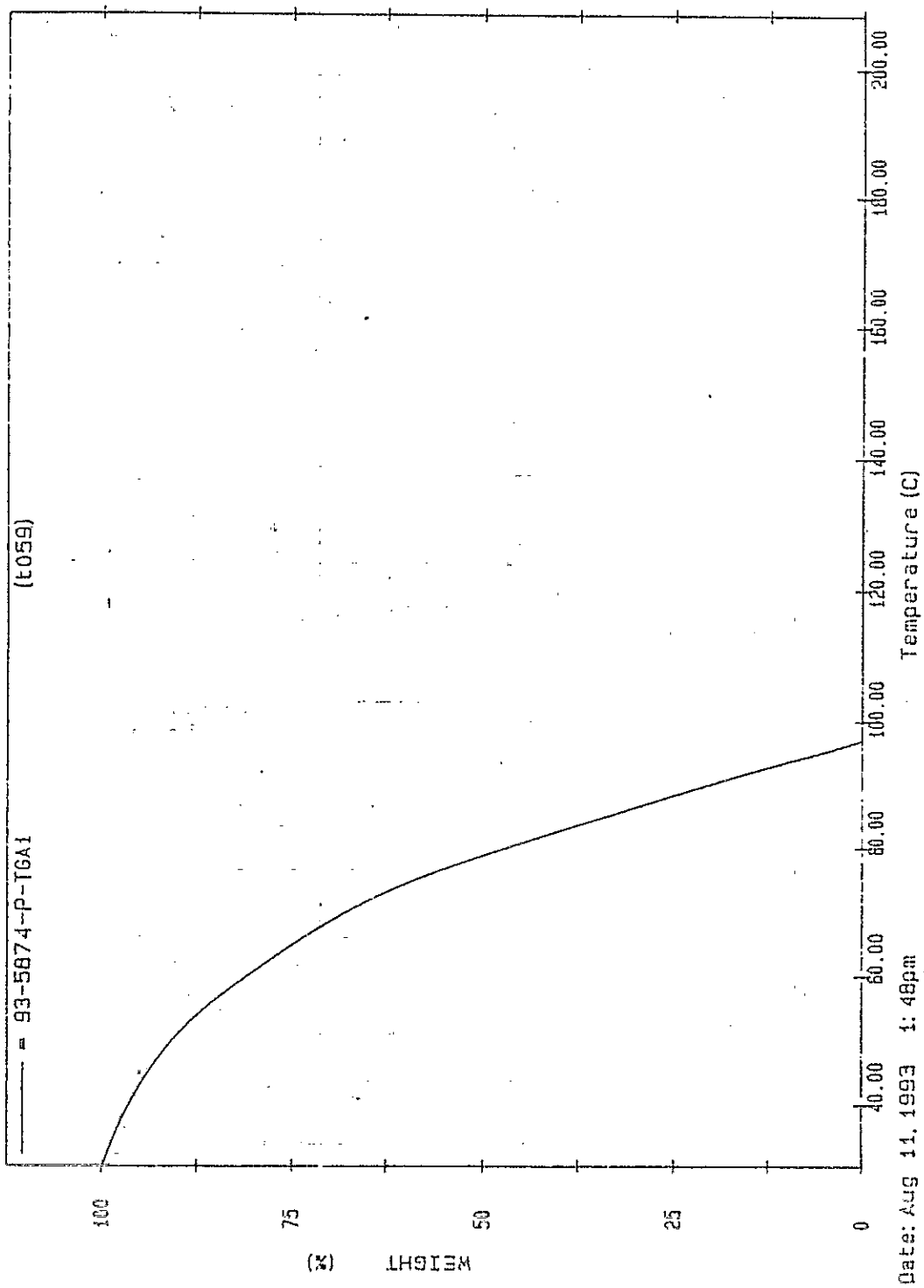
WHC-SD-WM-DP-052,
ADDENDUM 1 REV. 0

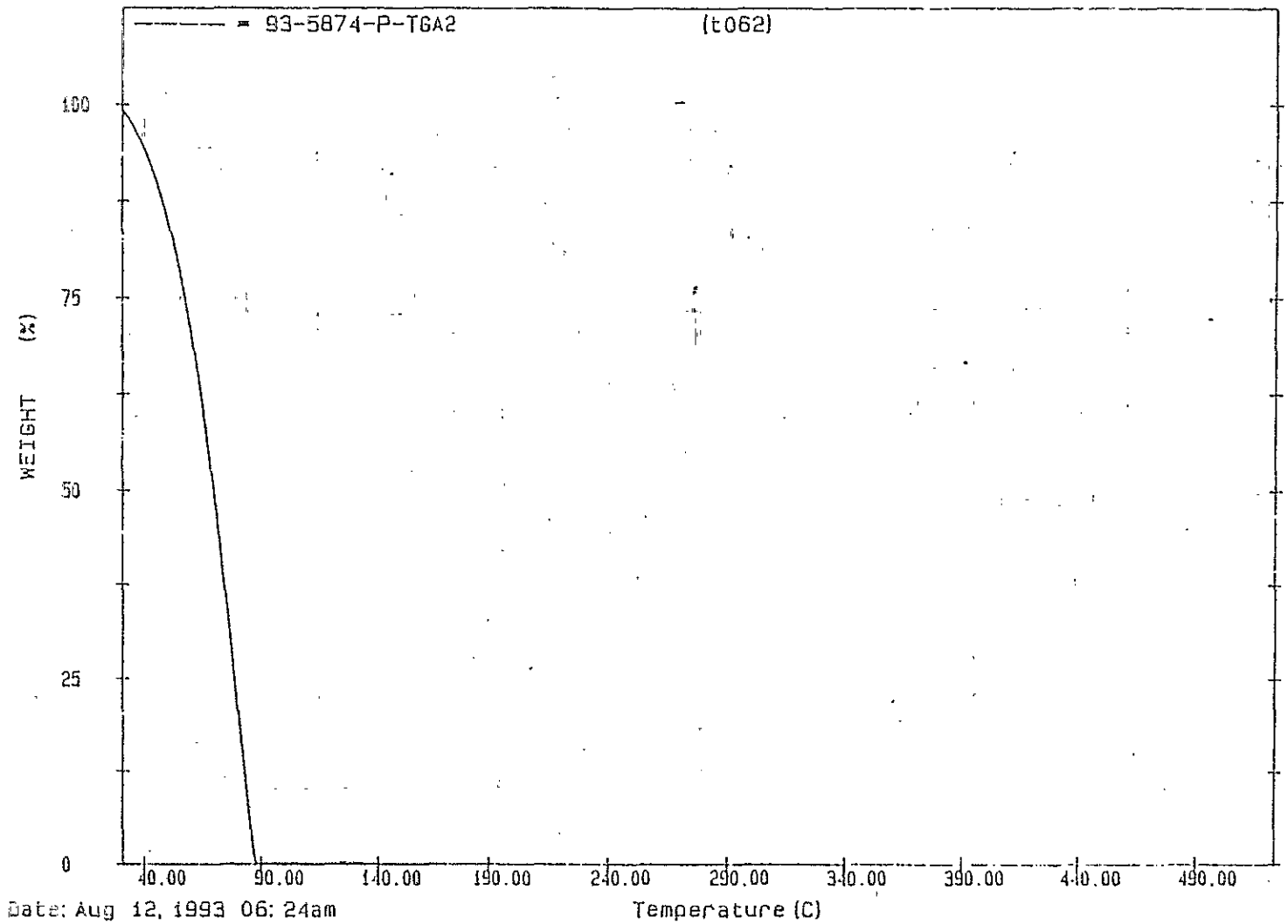
Perkin-Elmer TGA7

A-237

C09-010

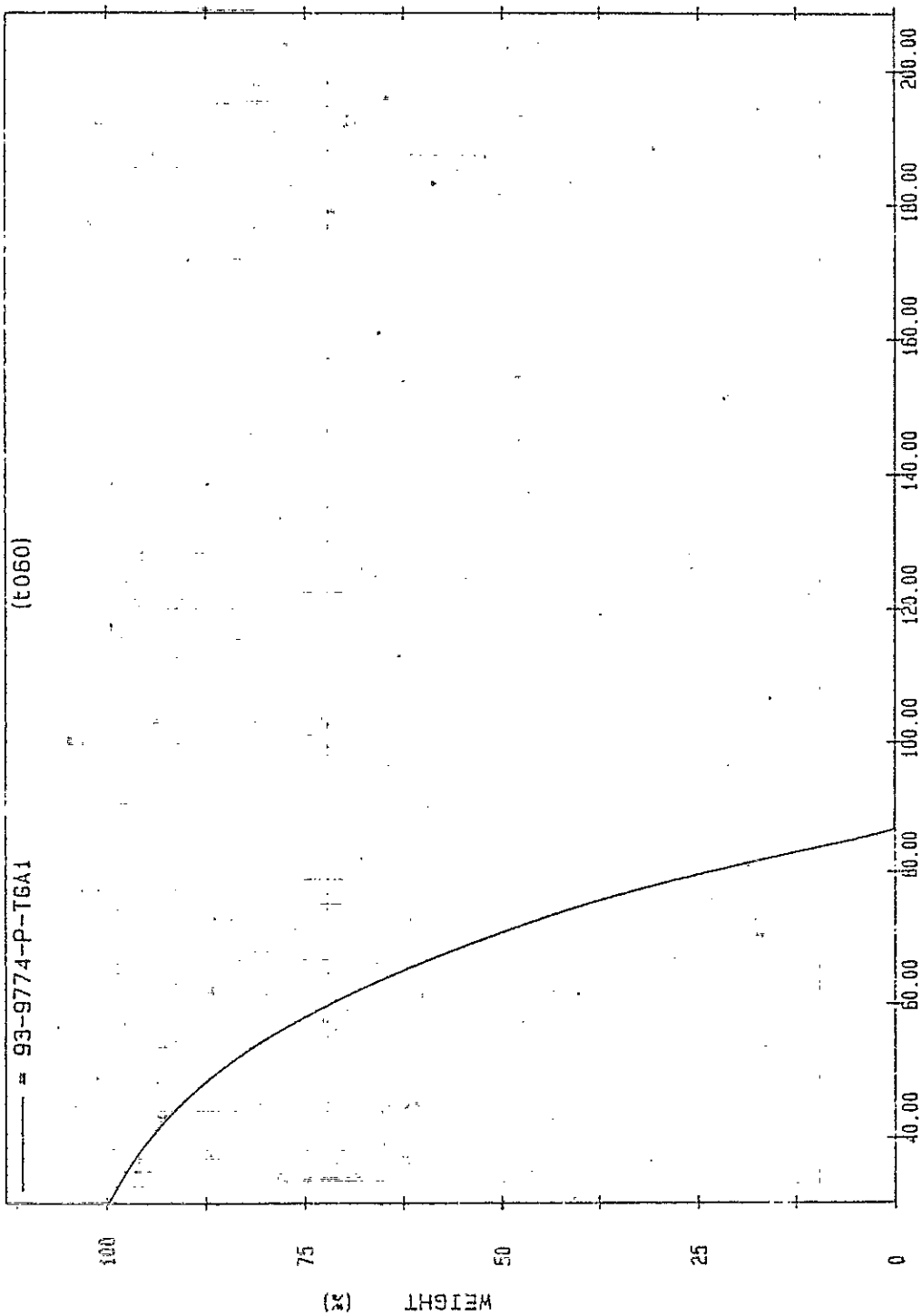
C00305

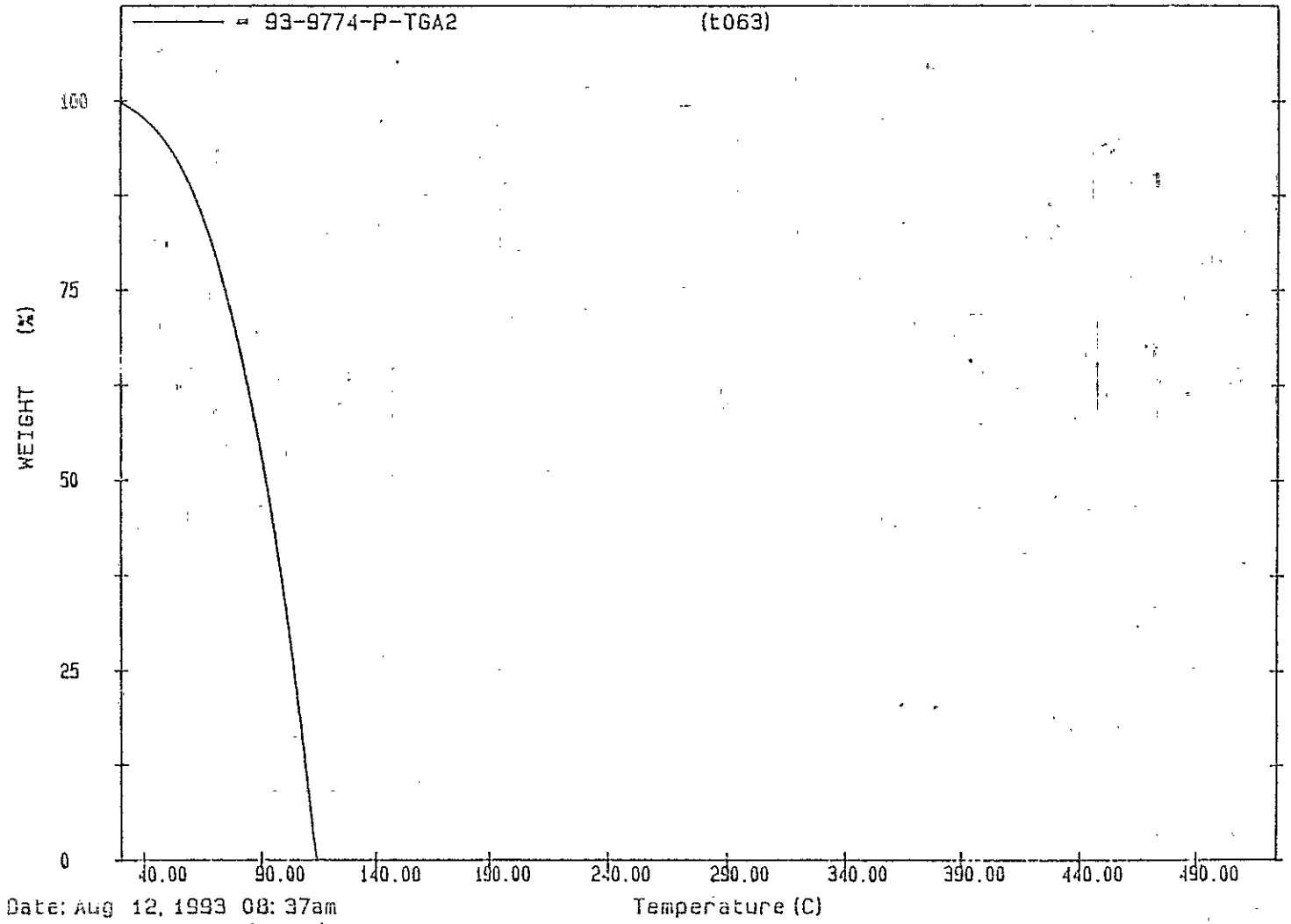

93-5874-P-TGA1
(t059)
WEIGHT (%)
100
75
50
25
0
40.00
60.00
80.00
100.00
120.00
140.00
160.00
180.00
200.00
Temperature (C)
Date: Aug 11, 1993 1:48pm
Scanning Rate: 2.0 C/min
Sample Wt: 25.708 mg Path: a:\
File: T059 ALS
Perkin-Elmer TGA7



Date: Aug 12, 1993 06:24am
Scanning Rate: 5.0 C/min
Sample Wt: 7.318 mg Path: a:\
File: T062 ALS

Perkin-Elmer TGA7


(t060)
93-9774-P-TGA1
WEIGHT (%)
100
75
50
25
0
40.00
60.00
80.00
100.00
120.00
140.00
160.00
180.00
200.00
Temperature (C)
Date: Aug 11, 1993 3:52pm
Scanning Rate: 2.0 C/min
Sample Wt: 18.091 mg Path: a:\
File: T060 ALS
Perkin-Elmer TGA7



Date: Aug 12, 1993 08:37am
Scanning Rate: 5.0 C/min
Sample Wt: 18.394 mg Path: a:\
File: T063 ALS

Perkin-Elmer TGA7

DATE TO QC: August 17, 1993

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

DATA QUALITY REVIEW

I have reviewed the following data for comoleteness and for compliance with project requirements.

Analyte - Total Dissolved Solids

Data Package/Report - Core 55

ACL Numbers - 93-08755-C1 93-08755-C2 93-08755-C3

Kristine J. Kuhl-Klinger
PNL ACL Quality Representative

8/25/93
Date

1A-222
C10-002

000210

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

TOTAL DISSOLVED SOLIDS DATA SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

CLIENT: TWC/S. G. McKINLEY WORK PACKAGE: M99592/20777 ASR/ARF/LOI/TI: TI-TWC-06

QA PLAN: ALO-003 IMPACT LEVEL: II PROCEDURE NUMBER: PNL-ALO-501

TANK T-102, CORE 55 SLUDGE 93-08755
SAMPLE IDENTIFICATION

ACL NUMBER	CLIENT IDENTIFICATION	TARE WEIGHT (G)	(A) SAMPLE WET WEIGHT PLUS TARE	(B) SAMPLE DRY WEIGHT PLUS TARE	TOTAL DISSOLVED SOLIDS
93-08755-C-1	C55-FIL	8.2143	13.1744	8.2191	.0048 g = 0.10%
93-08755-C-2	C55-FIL	8.3554	13.3545	8.3601	.0047 g = 0.09%
93-08755-C-3	C55-FIL	8.1570	13.1307	8.1572	.0002 g = <0.01%

TDS = $\frac{\text{B - TARE}}{\text{A - TARE}}$ X 100

DATE/TIME IN: 8/11/93 1245 OVEN TEMPERATURE: 104 °C

DATE/TIME OUT: 8/12/93 0130 OVEN TEMPERATURE: 105 °C

THERMOCOUPLE: ~~01862~~ SN6311 GS 8/13/93

BALANCE: CELL 2 (360-06-01-016) X

BALANCE: CELL 6 (362-06-01-038) ___

Analyst: [signature] Date: 8/12/93

Reviewer: [signature] Date: 8/13/93

C10-003
14-2223

ET2003

Battelle
Pacific Northwest Laboratories

Project Number 16021

Internal Distribution

File/LB

Date September 15, 1993

To Susan G. McKinley

From Ingrid Burgeson

WHC-SD-WM-DP-052
ADDENDUM 1 REV. 0

Subject Cyanide Results for T-102 Core 55 and Field and Hot Cell Blanks

Correspondence under Project 20777, Work Order M99573. The T-102 core 55 samples were digested in the Shielded Analytical Laboratory (SAL) using method PNL-ALO-285. Since high levels of cyanide were not expected in this tank, the samples were digested with some modifications: sections 3.8, 4.4 and 4.5 were not implemented. The samples were analyzed for cyanide on August 18, 1993 in the 325 building, room 313 utilizing a Lachet Autoanalyzer (WC36517) following the manufacturer's recommended procedure. The CRA solution level has not yet been determined so it was not analyzed. This issue has been addressed in DR#93-033. The cyanide instrument detection limit is 0.0017 µg/mL for liquids and 0.09 µg/g for solids. The method detection limit is 0.017 µg/mL for liquids and 0.90 µg/g for solids.

The three ICV/CCV verification standards had an average recovery of 98% with a deviation of 0.6%. The sample duplicate Relative Percent Difference was 13% for sample 93-08755. The other sample duplicate recoveries were not reported, because the analyte levels were less than the method detection limit. The spike recovery for sample 93-08755 was 81% and the spike control (spike blank) recovery was 88%.

Samples 93-05874, 93-09774 and 93-09804 were analyzed without digestion, thus the results in Table 2 represent the free cyanide in solution. The cyanide sample aliquots were consumed in the initial analysis; therefore; the remaining solution from the IC aliquot was digested and analyzed for total cyanide. The two ICV/CCV verification standards had an average recovery of 104%. The CCV/CCB analysis for this batch was performed after eleven samples had been analyzed; however, the sample results were not adversaly affected. There were no duplicate analyses, instead two of the samples were spiked. The spike recovery for sample 93-05874 was 104% and 124% for sample 93-09774. The spike control (spike blank) recovery was 102%. The digested blank sample results are reported in Table 3.

All analytical and quality control data are archived in the System Archive CN-325-313 File: CN081893 and CN090893.

ES4-1900-001 (10/89)

ICP Analyses -- Fusions Results

ICP analyses were performed on fusions prepared from Core 55 samples. The samples were prepared following procedure PNL-ALO-102, "Fusion of Hanford Tank Waste Solids" (KOH fusions in Ni crucibles), and analyzed following procedure PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry." The fusions and subsequent melt acid dissolutions were performed in the SAL and the digestates transferred to the Inorganic Analysis Group for subsequent ICP analyses. All ICP analyses were performed on the Jarrell-Ash ICP system. All interelement corrections for spectral interferences were performed on-line.

The fusion results for the composite sample, duplicate, and blank are reported along with the post spike QC results. Core 55 composite samples was analyzed at both a 2x and 10x dilution with the corresponding percent difference (%D) used to indicate potential matrix interference; percent differences exceeding 10% are suspect provided the 10x dilution result is greater than five times the MDL. The RPD for duplicate analyses is shown, and the flag "*" is used to indicate when the RPD has exceeded 20% and the quantitated results exceed the MDL. An estimate of the sample detection limit can be obtained by multiplying the analyte's "IDL" value by the appropriate sample "Dil Factor." It should be noted that the process blank analyzed has not been subtracted from the reported sample results.

Core 55 Composite (93-08755-H): The ICP results for the core composite show the major analytes to be Al, Fe, and Na; totalling approximately 36 percent, wet weight. The comparison between the fusion results and acid digestion results is reasonably good for Fe and Na; however, Al results for the acid digestion is about 50% that of the fusion result. The RPDs for the major concentration analytes were poor, indicating difficulty in obtaining representative sample for the fusion preparation (note: fusion procedure uses only 0.2 g of material for dissolution which may add significantly to the apparent heterogeneity). The processing blank shows no analyte concentrations above the MDL and the post spike analysis shows good recovery for all analytes.

Analytical QC Associated with Fusion Analyses: Four analyte failures involving analytical QC samples SSTMCV and SSTICSAB were observed. Silver, arsenic, and cadmium failed on the SSTMCV standard and arsenic, cadmium and zirconium failed on the SSTICSAB. The SSTMVC standard failed Ag at a concentration exactly twice the expected concentration; the ICV and SSTICSAB standards quantified Ag accurately and initial indications are that the SSTMCV standard preparation for Ag is in error. Cadmium marginally failed the SSTMCV and SSTICSAB; demonstrating slightly high results. The Cd results which are above the IDL but less than the MDL may be an artifact of the high bias; the problem is being investigated. Zirconium failed on the SSTICSAB standard and appears to be from a loss due to phosphate precipitation. The Zr failure is not considered to significantly affect the sample results since no Zr was detected in the samples, the P in the samples is very low, and the SSTMCV demonstrated excellent Zr recovery. The arsenic channel continues to demonstrate erratic behavior and the all arsenic results are considered unusable.

ICP Analyses -- Acidified "Blank" Results

ICP analyses were performed on water blanks (i.e., T-102 Field Blank, HLRF Hot Cell Blank, and HRLF DIW) associated with the processing of T-102 Core 55. The water samples were acidified with HNO_3 and then analyzed following procedure PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry." The acidifications were performed by SAL staff and the sample aliquots transferred to the Inorganic Analysis Group for subsequent ICP analyses. All ICP analyses were performed on a Jarrell-Ash ICP system. All interelement corrections for spectral interferences were performed on-line.

The water blanks were processed and analyzed as a single batch. All sample were analyzed at 1X and since no analytes except Na were found above 5 times the MDL, no serial dilutions were performed. The RPD for the duplicate analyses is shown, and the flag "*" is used to indicate when the RPD has exceeded 20% and the quantitated results exceed the MDL. An estimate of the sample detection limit can be obtained by multiplying the analyte's "IDL" value by the appropriate sample "Dil Factor"; note that at 1X the sample

detection limit is the IDL. Since these samples are "blanks" and are analyzed directly, no process blank has been analyzed with these samples.

HLRF Hot Cell Blank (93-09774): The only analytes detected above the MDL are Na and Ca, being about 5 and 0.5 µg/mL, respectively. A partial suite of sample QC (i.e., duplicate and pre-spike) was performed. The RPDs for Na and Ca are very good, as would be expected for duplicate water analyses. Spikes added to the sample by SAL demonstrated excellent recovery, ranging from 92% to 110% (Note: Only part A of the spiking solution was added to the HLRF Hot Cell Blank; therefore, As and Se are not present). The post spike for the fusion samples was prepared and analyzed with the water blank samples and shows good recovery for all analytes.

HLRF DIW (93-09804): There were no analytes detected above the MDL; therefore, the DIW used for the HLRF Hot Cell Blank should not contribute significantly to the Na and Ca concentrations observed in the Hot Cell Blank.

T-102 Field Blank (93-05874): The only analytes detected above the MDL are B, Ca, Na, and Si. The Ca and Na concentrations are similar to those found in the HLRF Hot Cell Blank. The B and Si appear to be unique to the Field Blank (i.e., relative to other water blanks analyzed). The RPDs for the analytes above the MDL are excellent; typical for duplicate, low concentration water samples.

Analytical QC Associated with "Blank" Analyses: Six analyte failures involving analytical QC samples SSTMCV and SSTICSAB were observed. Bismuth and silicon failed on the "old" SSTMCV standard; however, the "new" SSTMCV standard proved to analyze these analytes without any problem. The "new" SSTMVC standard failed Ag at a concentration exactly twice the expected concentration; the ICV and ICSAB Standards quantified Ag accurately and initial indications are that the SSTMCV standard preparation for Ag is in error. Cadmium marginally failed both the "old" and "new" SSTMCV standard as well as the SSTICSAB; demonstrating slightly high results -- this problem is being investigated. However, the problem is not considered to significantly affect the water blank sample's Cd results since no Cd was detected in the water blanks above the MDL. Zirconium failed on the SSTICSAB standard and

appears to be from a loss due to phosphate precipitation. The Zr failure is not considered to significantly affect the sample results since no Zr was detected samples, the P in the samples is very low, and the SSTMCV demonstrated excellent Zr recovery. The arsenic channel continues to demonstrate erratic behavior and the all arsenic results are considered unusable.

9/10/93

File: fus-wat.102

NOTE: NO CRI MDL STANDARD ANALYED WITH THIS DATA; SEE DR-93-033 9/13/93

Homogenization Tests: Core 55 Composite (93-08755-H Top/Bottom): The KOH fusion results for the core composite homogenization test shows no "statistical" difference (based on a mean Student "t") between the top and the bottom sample results; this is primarily because the analytical variability between all samples (top, top duplicate, bottom, and bottom duplicate) is very large. A significant number of the RPDs (comparing all four samples) are higher than normal (i.e., 10-60% verses <10%), indicating a poor homogenization. Particularly disturbing are the Fe and Na results which, if evaluated as a percent difference calculated by dividing the largest difference between the samples by the average of the four samples, shows differences in the range of 32% to 55%. Aluminum also demonstrated a higher than normal difference of about 10%. Based on these results, attempts were made to improve homogenization by reblending; however, due to the lack of sample, no further ICP homogenization tests were performed. Difficulties in obtaining quality duplicate samplings compromise the accuracy of the full suite of characterization analyses performed, as well as adversely affecting the ability to obtain good pre-digestion spike recoveries.

It should be noted that, that in accord with the governing QAPjP, no sample QC was performed other than the duplicate analyses for the top and bottom samples and a fusion processing blank. Two analyte failures involving analytical QC samples (i.e., SSTMCV and SSTICSA8) were observed. Bismuth failed on all runs for the SSTMCV standard; however, this is not considered to significantly affect the sample results since no Bi was detected in the samples and the Bi in the SSTICSA8 standard demonstrated excellent recovery (i.e., averaging 95% recovery at 201.5 µg/mL). Zirconium failed on both runs of the SSTICSA8 standard and appears to be from a loss due to phosphate precipitation. The Zr failure is not considered to significantly affect the sample results since no Zr was detected samples, the P in the samples is very low, and the SSTMCV demonstrated excellent Zr recovery (i.e., averaging about 102% recovery at 0.5 µg/mL).

8/31/97

ICP Analyses -- Acid Digestion Results

ICP analyses were performed on acid digestions prepared from Core 55 samples material. The core composite samples were prepared following procedure PNL-ALO-101, "Acid Digestion for Metals Analysis" (i.e., HNO_3/HCl), and analyzed following procedure PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry." The sample acid digestions were performed in the SAL and the digestates transferred to the Inorganic Analysis Group for subsequent ICP analyses. All ICP analyses were performed on a Jarrell-Ash ICP system with interelement corrections for spectral interferences being performed on-line.

The acid digestion results for the composite sample, duplicate, and blank are reported along with the associated sample QC results. Core 55 composite leachate was analyzed at both a 2x and 10x dilution with the corresponding percent difference (%D) used to indicate potential matrix interference; percent differences exceeding 10% are suspect provided the 10x dilution result is greater than five times the MDL. The RPD for duplicate analyses is shown, and the flag "*" is used to indicate when the RPD has exceeded 20% and the quantitated results exceed the MDL. An estimate of the sample detection limit can be obtained by multiplying the analyte's "IDL" value by the appropriate sample "Dil Factor." It should be noted that the process blank analyzed has not been subtracted from the reported sample results. However, processing blank results >IDL are subtracted from the Blank Spike control prior to determining the percent spike recovery. Also, no CRI MDL standard was analyzed; see deficiency report DR-93-033.

The acid digestion results for core composites correlate reasonably well with those from the fusion preparations except that Al and Si are significantly lower, as would be expected for an acid digestion/leach. The percent difference between a 2x and 10x dilutions for both the sample and duplicate is very good, indicating that the instrument results obtained on the digestion solutions are reliable. The RPD values for Al, Fe, and Na are acceptable (i.e., <20%); indicating "adequate" homogenization, subsampling, and analytical precision. Three analytes demonstrated blank concentrations above the MDL; B, Ca, and Na; the B and Ca "contamination" significantly

impact the reliability of the reported results. The sample and duplicate results indicate that the primary analytes are Al, Fe, and Na; totalling approximately 20 percent, wet weight. Also of interest is the Cr and Pb, since if moderately TCLP leachable, either analyte would classify the tank material as toxic. Silicon, which is also an moderate concentration analyte as indicated from the fusion analysis, is not typically completely solubilized by the acid digestion procedure used.

The full suite of QC (i.e., duplicate, pre-spike, and post-spike) was performed. The percent recoveries for the spike "control" (i.e., the blank spike) are reasonably good with most recoveries being between 80-120%; except Bi and K which recovered at 69% and 73%, respectively. The percent recoveries for the spiked samples are good with recoveries being within acceptable limits for most category A and B analytes for which spiking was performed; silicon is the noticable exception, recovering at only 39%. For a few spikes, recovery is meaningless since the spike is less than 25% of the sample's measured concentration (as indicated by "N" Sok flag). The pre-digestion spike additions for Al, Cr, Fe, Mn, and Na were insufficient for recovery quantitation. All post-digestion spikes, except Si, met the 75%-125% acceptance criteria.

Four analyte failures involving analytical QC samples (i.e., SSTMCV and SSTICSAB) were observed. Arsenic, bismuth and silicon failed on runs of the SSTMCV standard. The Bi in the SSTMCV routinely fails and is considered to be an standard preparation error (Note: "new standard" make-up is in progress); however, other standards containing Bi validate the usability of the sample results (i.e., the SSTICSAB and MCVA). Silicon showed instability during the analytical runs and the reported results are considered to be questionable (i.e., a very large uncertainty range). Zirconium failed on both runs of the SSTICSAB standard and appears to be from a loss due to phosphate precipitation. The Zr failure is not considered to significantly affect the sample results since no Zr was detected samples, the recovery of the pre-spike, blank spike, and post-spike are very good, and the SSTMCV demonstrated good Zr recovery. The arsenic failure in the SSTICSAB and SSTMCV standard is attributed to a faulty As channel and will require instrument maintenance. Arsenic results are considered erratic and unusable.

ICP Analyses -- Water Leach Results

ICP analyses were performed on water leaches prepared from Core 55 samples material. The core composite samples were leached following procedure PNL-ALO-103, "Water Leach of Sludges, Soils, and Other Solid Samples," and then analyzed following procedure PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry."

The water leaches were performed in the SAL and the sample aliquots transferred to the Inorganic Analysis Group for subsequent ICP analyses. All ICP analyses were performed on a Jarrell-Ash ICP system. All interelement corrections for spectral interferences were performed on-line.

The water leach results for the composite sample, duplicate, and blank are reported along with the associated sample QC results. Core 55 Composite leachate was analyzed at both a 2x and 10x dilution with the corresponding percent difference (%D) used to indicate potential matrix interference; percent differences exceeding 10% are suspect for reported values above five times the MDL. The RPD for duplicate analyses is shown, and the 20%-flag (*) is used to indicate when the RPD has exceeded 20% and the quantitated results exceed the MDL. An estimate of the sample detection limit can be obtained by multiplying each analyte's "IDL" value by the appropriate sample "Dil Factor." It should be noted that the process blank has not been subtracted from the reported sample results.

The major water soluble analyte appears to be Na, with very minor contributions from Al, Cr, Fe and P. The components measured by the ICP on the water leach account for only about three percent of the total sample wet weight. This water soluble fraction represents about a tenth of the wet weight fraction of analytes measured from the fusion preparation, which is considered to be a complete dissolution. The sample and duplicate RPD is considered very good for the Na results and adequate for the remaining analytes at low concentrations.

The full suite of sample QC (i.e., duplicate, pre-spike, and post-spike) was performed. The percent recoveries for the spike "control" (i.e., blank

spike) are reasonably good with all recoveries being between 80-120%; except Bi which shows a high bias (i.e., 167% recovery). The percent recoveries for the spiked sample are generally very poor with recoveries ranging from "not detectable" to well in excess of 200%. The primary explanation for this phenomenon is that the high acid spike solution has been added to the sample during the leaching process; this changes the leaching characteristics of the leach and 1) extracts higher concentrations of some analytes and 2) leads to precipitation of spiking analytes due to a significant pH change. For a few spikes, recovery is meaningless since the spike is less than 25% of the sample's measured concentration (as indicated by "N" Spk flag); the pre-digestion spikes additions for Cr and Na were insufficient for recovery quantitation. The post-digestion spike recovered very well for all analytes (except Si which was slightly out of the 75-125% recovery limit) for those analytes which were at spiking concentrations exceeding 25% of the sample's concentration.

Four analyte failures involving analytical QC samples (i.e., SSTMCV and SSTICSAB) were observed. Arsenic, bismuth and Silicon failed on all runs for the SSTMCV standard; to verify control, additional single element standards were analyzed and met recovery criteria. However, it should be noted that the Bi in the SSTMCV routinely fails and is considered to be an standard preparation error; other standards containing Bi validate the usability of the sample results. Silicon showed instability during the analytical runs and the reported results are considered to be questionable (i.e., a very large uncertainty range). Zirconium failed on both runs of the SSTICSAB standard and appears to be from a loss due to phosphate precipitation. The Zr failure is not considered to significantly affect the sample results since no Zr was detected samples, the recovery of the blank spike and post spike are very good, and the SSTMCV demonstrated good Zr recovery. The arsenic failure in the SSTICSAB standard is attributed to a faulty As channel (as is the As failure in the SSTMCV standard) and will require instrument maintenance. Arsenic results are considered erratic and unusable.

File: Water.tm

No CRI MDL std analyzed; See DR-93-033

9/9/93

Battelle
Pacific Northwest Laboratories

Project Number _____

Internal Distribution

File/LB

Date September 3, 1993

To SST Project Management Office

From DL Baldwin DL Baldwin

Subject Direct TOC/TIC/TC for T-102 Core 55 Sludge Samples and Hot Cell Blanks/Field Blanks/ DIW Blank

This work is done by the hot persulfate oxidation method, Test Procedure PNL-ALO-381, rev. 0, "Determination of TC, TOC, and TIC in Radioactive Liquids, Soils and Sludges by Hot Persulfate Method". The M&TE No. for the carbon measurements is WC01713, the balance M&TE No. is 360-06-01-016. The data is located on the accompanying data sheets, review reports or on file in the ALO Records Office. TOC standard used is alpha-d-glucose, Kodak lot# B1F, and the TIC standard is $CaCO_3$, lot N262. Both materials are used in solid form for system standards as well as matrix spikes.

Narrative: The analysis of the Hot Cell blanks, Field blanks and DIW blanks was done in one batch on 8/18/93. The analysis of the core sample, dup, and matrix spike, 93-08755-J1, J2, and J4, was done in one extended batch over two days, 8/19/93-8/20/93. All of the QC came within limits except for the TOC portion of the original matrix spike, at 60%, and the third system standard TIC and TOC, at 61% and 62%. Therefore, a fourth system standard was successfully done on the first day, and a blank, system standard and matrix spike were successfully repeated on the second day. But since the original TIC matrix spike, which is run before the TOC spike, came in within limits at 86%, the previous sample analyses were considered satisfactory.

The QC values were as follows. TIC/TOC system standards, excluding the out-of-limit values, gave average recoveries from 92% to 98% for TIC and 91% to 95% for TOC. System blank levels were fairly consistent throughout the batch. The one Matrix Spike gave poor results of 60% for TOC, but all other Matrix Spikes were within limits, with TIC spikes from 86% to 117% and TOC spikes from 103% to 106%. The RPD's for sample 93-08755 were 15% for TIC, but for TOC were out of limits at 28%. There is no experimental explanation for this high RPD, possibly indicating inhomogeniety for this sample. The RPD's shown were rounded to the nearest integer, but were calculated based upon the full displayed digits in the spreadsheet review reports, so there may be some rounding error. The estimated precision is ±10% and the estimated accuracy is ±15%.

The Hot Cell blanks and Field blanks showed very low levels of TIC and TOC. An MDL was established based on historical system blank data, using 3x the pooled SD of the most recent 12 sample batch blank results.

BS-1900-401 (10/89)

This resulted in samole results of ND-6 ug/ml TIC, ND-11 ug/ml for TOC, and ND-18 ug/ml TC, compared with the DIW blank of ND-3 ug/ml TIC, 7-9 ug/ml TOC and 7-11 ug/ml for TC.

ALO No.	Sample wt (g)	TIC (ug/g)	RPD	TOC (ug/g)	RPD	TC (ug/g)	RPD
93-08755 -J1	0.1236	2280		520		2800	
93-08755 -J2	0.1992	2660	15	680	28	3350	18
93-08755 -J4	0.1948	86% recovery		60% recovery			
93-08755 -J4 (repeat)	0.1594	117% recovery		106% recovery			

ALO No.	Sample vol(ml)	TIC (ug/ml)	TOC (ug/ml)	TC (ug/ml)
93-05874 -P1 H C Blk	5.0021	6	11	18
93-05874 -P2 H C Blk	5.0021	4	11	15
93-09774 -P1 F Blk	4.0028	3	ND	3
93-09774 -P2 F Blk	4.0028	ND	ND	ND
93-09774 -P4 F B MS	4.0028	98% Recovery	103% Recovery	
93-09804 -P1 DIW Blk	5.0021	3	9	11
93-09804 -P2 DIW Blk	5.0021	ND	7	7

Notes:

(1) Only TIC and TOC are actually measured. The TC is the sum of TIC and TOC.

(2) Percent recovery is determined for TIC and TOC using the respective standards, CaCO3 or glucose, and all sample results are corrected for percent recovery. All results are blank-corrected.

(3) The reported results have been rounded to two or three significant places, so some may slightly disagree with the spreadsheet review report. The RPD's were rounded to the nearest integer, but were calculated based upon the full displayed digits in the spreadsheet review report. No RPD's are reported for the blanks due to the low levels.

Concur by: _____ Date: 9/3/93

File TOC-PER.T2
System File: TOC083193

Battelle
Pacific Northwest Laboratories

Project Number _____

Internal Distribution

System File/L8

Date September 14, 1993

To SST Project Management Office

From DL Baldwin

Subject TC/TOC/TIC (Water Leach) Results for Tank T-102 Core 55

This work is done by the UV-catalyzed persulfate/NDIR method, Test Procedure PNL-ALO-382, Rev. 0, "Solutions Analysis: Carbon", using the Dohrmann DC-80 Total Organic Carbon Analyzer. The M&TE No. for the carbon measurements is WA64102, M&TE No. for the balance is 362-06-01-046. The data is located in the ALO Records Office System File. TC standard used is potassium acid phthalate, lot# 52809, and the TIC standard is sodium carbonate, lot# 52815.

Narrative: This work was done in one batch on 8/10/93. There were no apparent outliers. The QC came within established limits, except for the Method Blank. The TC matrix spike was 102% and the TIC matrix spike was 111%. The RPD's were excellent at 4.1%, 1.5% and 2.6%, respectively for TC, TOC and TIC. The method blank for TC and TOC was 80 ug/g, both above the stated MDL for this work of 50 ug/g, as noted in Note (4) below. This above-MDL method blank, though low, indicates the presence of TOC from SAL handling. No -C4 matrix spike sample was received from the SAL for analysis. The matrix spike shown for -C1 is an analyst-added spike added to the laboratory-diluted sample. Precision and accuracy for this method are estimated at ±10% and ±15%, respectively. The units are ug/g, based on the weight of the original sludge material.

ALO Number	Sample ID	TC (ug/g)	RPD %	TOC (ug/g)	RPD %	TIC (ug/g)	RPD %
93-08755 C1	C55-Smpl	4150		650		3500	
	Matrix Spike	102%				111%	
93-08755 C2	C55-Dupl	3980	4.1	660	1.5	3410	2.6
93-08755 C3	C55-MB	80		80		ND	

1) Only TC and TIC are actually measured. TOC is found by the subtraction of TIC from TC.
2) Matrix spike recovery was determined for TC and TIC, in duplicate, using the respective standards.
3) The C55-Sample and C55-duplicate were each analyzed in duplicate. Result shown is the average.
4) An MDL was determined by the pooled SD of five batch blanks. MDL is set equal to 10x pooled SD. This is set equal to 0.5 ug/ml, prior to any

554-1993-001 (10.93)

14-236

D09-016

000224

process dilution factor, for both TC and TIC. Applying the 100x process dilution factor to C55-MB, the TIC ND is equal to <50 ug/g.

Concur by: [signature] Date: 9/14/93
Disk File: UV-TOC.22 System File: TC081093

DR-93-033 Addressed absence of matrix spike & spike control.
[signature]
9/15/93

Battelle
Pacific Northwest Laboratories

Project Number _____

Internal Distribution

Date September 2, 1993

To SG McKinley

From MC Burt

Subject Determination of NH_3-N in SST Samples

WHC-SD-WM-DP-052.
ADDENDUM 1 REV. 0

Leach solutions and blanks from T-102, Core 55 received for analysis were analyzed according to Procedure PNL-ALO-226. Data is recorded in the system file in Rm. 406 Bldg. 325 as file NH3930902. M&TE used were Corning Meter Model 240 and WB76707 Mettler balance.

Analytical spikes were added to each sample and blank solution after the initial measurement was made. The spike recoveries on all analyses were within the prescribed range of 80-120%. These samples were all measured at the low end of the calibration curve where very small differences in measured values have a large effect on quantitated values. This could account for the difference between duplicates on sample 93-08755-C-1 and C-2. There was insufficient sample for a reanalysis. Because all analytes are less than ten times IDL no RPD values are reported.

The system detection limit (defined as IDL) is set at the lowest calibration point of 0.05 ug NH_3-N/mL as the electrode response is very non-reproducible at lower values. The method detection limit (MDL) is by definition 0.5 ug/mL (10 times IDL).

ALO Number	Cust. Ident.	NH_3-N, ug/g	Spike Rec., %
93-08755-C-1	Tank T-102 Core 55	(27.2)	100
93-08755-C-2	Tank T-102 Core 55	ND	98
93-08755-C-3	Tank T-102 Core 55	ND	96
		NH_3-N, ug/mL	
93-05874-R-1	Tank T-102 FIELD BLANK	ND	90
93-05874-R-2	Tank T-102 FIELD BLANK	ND	90
93-09774-R-1	Tank T-102 HLRF HC BLK	ND	90
93-09774-R-2	Tank T-102 HLRF HC BLK	ND	90
93-09804-R-1	Tank T-102 HLRF DIW	ND	90
93-09804-R-2	Tank T-102 HLRF DIW	ND	90

A nominal sample weight of 1.5g is used to calculate the process blank in mg/Kg. Actual sample results are based on sample weights and volumes as received.

If there are questions regarding the results please contact MC Burt on 376-3762.

Concur: [signature]

Note: No Pre-leach spiking was performed. See DR-93-033. [signature] 4/9/93

Battelle
Pacific Northwest Laboratories

Project Number _____

Internal Distribution

Date September 1, 1993

To SG McKinley

From MC Burt

Subject Titration for Hydroxyl Ion

WHC-SD-WM-DP-052-
ADDENDUM 1 REV. 0

The water leach solutions from T-102, Core 55 and the hot cell blank were analyzed according to PNL-ALO-228. M&TE used was Titroprocessor 636, WB76843, Digital Buret, WB76839 and balance WA83665. Data is recorded in LRB52846 pp. 51-52.

Because the pH of the solutions were very close to neutral a standard titrant was prepared at 0.02N. Even with the low concentration titrant, no titration could be performed on 93-05874/Field Blank, 93-09774/Hot Cell Blank, and 93-09804/HLRF DIW and the sample blank due to the lack of a titratable constituent. The entire 5 mL portions of 93-09774-P1 and -P2 were combined for titration and 2 mL of 93-08755-M3 were titrated, both with no result.

The procedure determines basicity resulting from hydroxyl, carbonate and bicarbonate ions. There was no hydoxyl ion detectable in sample solutions 93-08755-M1 and -M2, the basicity being attributable to carbonate and bicarbonate. A 'less than' value for hydroxyl may be determined by making two assumptions. The first is that 0.100 mL of titrant is required to reach EP 1 and second, that the result from EP 2 is less than two times the result from EP 1. This results in a value of <37 ugOH-/g of sample taken. There was insufficient sample to perform the 1:1 extraction as specified, and a 1:5 extraction was used for the titrations.

Because carbonate and bicarbonate are not reported no standard for them was analyzed. Standard NaOH #C-110 at 0.1047N was successfully analyzed at 0.1044N with this sample group.

If there are any questions regarding this result please contact me on 376-3762.

MC Burt, Sr. Res.Scientist
Analytical Chemistry Laboratory

Concur

/A-240

D10-003

000228

Ion Chromatography

The samples in this group were analyzed using procedure PNL-ALO-212, in accord with EPA Method 300.0. The sample preparation and analysis were performed in the 325 building in the 300 area by members of the ACL-Inorganic Analysis staff. Samples analyzed were 93-08755/T-102, Core 55, 93-05874/T-102 Field Blank, 93-09774/T-102 HLRF Hot Cell Blank, and 93-09804/T-102 HLRF DIW.

Data presentation (see Spreadsheets, and the notes below)

Each anion has been listed on a separate page with sample, sample duplicate, matrix spike and duplicate matrix spike, and spike control information for the Core sample. Separate sheets give data for the various blank samples and the MDL standard.

RPD values for duplicate analyses are reported together with spike recoveries for spiked samples and spike control standard recoveries. All analyte values, spike levels, and recoveries for the Core sample were based on weights used.

The control standard for all anions has been defined as the spike control.

MDL and IDL values

Method Detection Limits (MDL) have been established at the lowest calibration level of the procedure and Instrument Detection Limits (IDL) have been defined as one half of that level. Documentation has been provided to the Project Manager.

Accuracy and Precision in IC results

The IC anions analysis system has been calibrated with six calibration standards ranging from 0.25-7.5 ppm for the halides (F,Cl) and 0.5 - 30 ppm for the oxy-anions (NO2, NO3, PO4, SO4). The accuracy of the calibration was checked by analyzing within ±10% three independently derived verification standards at approximately 17%, 50%, and 83% of the calibration range maximum. Additionally, a standard prepared from the same source materials as the verification standards at the defined MDL was analyzed within the ±30% acceptance criteria. A spike check standard at 2 ppm halides and 5 ppm for oxy-anions at the time of injection was also analyzed within the ±10% acceptance criteria.

The accuracy of reported values between 20-80% of the calibration maximum has been estimated to be ±10%, unless otherwise noted in the Problems section of this report. The accuracy decreases and is estimated to be ±30% at the method detection limit (MDL) and may be 100% at the instrument detection limit (IDL).

Quality Control.

The criterion that the MDL standard for the anions of interest are quantitated within ±30% has been met.

RPD values for all analytes on the T-102, Core 55 samples were within the 20% acceptance limits. RPD values are not reported for the Field Blank, HLRF Hot Cell Blank, and HLRF DIW because analyte levels are less than 10 times IDL. The duplicate (-O-2) of sample 93-05874/T102 Field Blank has a higher nitrate level than the -O-1, and appears to have been contaminated. Because the -O-2 value is greater than 10 times IDL an RPD is reported, however it is higher than the ±20% criteria.

Matrix spike recoveries for all analytes on 93-09774/HLRF Hot Cell Blank were within ±20% acceptable limits.

Matrix spike recoveries for all analytes on sample 93-08755/T-102, Core 55 were within ±20% acceptance limits.

Problems

Recoveries on the spike control sample (93-08755-C-5) are all high, with only chloride and nitrite being within the ±20% acceptance limits. Sample 93-08755-C-4 and 93-09774-O-4/T-102 HLRF Hot Cell Blank were spiked with the same spiking solution and recoveries for all analytes were within acceptable limits which confirms that the spike solution is good. A comparison of quantitated values for all analytes on 93-08755-C-4 (except NO3 which is too high) at 10X and 1X shows good agreement which eliminates an analytical pipetting error. The average recovery for all analytes is 125±5% and this level of reproducibility would indicate a sample make up error rather than analytical error.

Although spike control recoveries of four analytes are outside the established recovery window the data is reported because system operation was satisfactory and all other criteria were met.

Other problems encountered in this run were matrix interferences at the fluoride peak on 93-05875/T-102, Core 55 which could bias the quantitated value an estimated 30-50% high. A matrix effect was noted on the chloride peak of sample 93-09774/T-102 HLRF Hot Cell Blank. The peak was slightly narrower than normal and may cause quantitation to be slightly low, however the effect is minimal.

DR-93-033 Addresses the issue of MDL/IDL. Letter defining values attached.
[signature]
9/14/93

[signature] 9/8/93

[signature] 9/8/93

Battelle
Pacific Northwest Laboratories

Project Number _____

Internal Distribution

Date August 30, 1993

cc: K.J. Kuhl-Klinger
T.L. Ehlert

To S.G. McKinley

From M.C. Burt

Subject IDL/MDL Determination for Ion Chromatography, Proc. ALO-212

As per instructions in ALO-003 Rev. 1 (and referenced 40 CFR 136,App. B), seven replicate measurements of a spiked blank have been measured on IC System 3 at different concentration levels on two separate days. The resulting data is presented on accompanying sheets.

The instrument detection limit (IDL) is calculated by multiplying the standard deviation (SD) by three, and the method detection limit (MDL) is defined as ten times the IDL.

The IC system has a rather narrow calibration range (0.25-7.5 ppm halides, and 0.5-30 ppm oxy-anions) and to extend the range could require changing method parameters which is not deemed desirable.

The first run on 7/21/93 measured replicates of a single solution at concentrations less than the lowest calibration standard level. Based on the above definitions, the resulting calculated MDL's are lower than the lowest calibration levels, except for chloride. Phosphate was not quantitated at the indicated level. It should also be noted that accuracy, as denoted by %Rec., was quite poor at the level measured.

A second run was made on 8/17/93 using standards at and slightly above the low calibration level and which were individually diluted rather than multiple runs of a single solution. Again, because precision of measurement is so good, MDL values less than low calibration level were obtained (except for fluoride which was at the low calibration level). This run indicated an MDL for phosphate at the same level which did not quantitate on the previous run. Accuracy (%Rec) was very good at the higher concentration level.

To accept MDL values below the lowest calibration level would require reporting data outside the calibration range of the method. This is poor analytical practice and would require reporting suspect data due to the poor accuracy below the low calibration levels. As indicated by the data, and because accuracy seriously degrades below the calibration range it would be difficult to reproducibly (and acceptably) measure a standard at the calculated MDL.

Based on these observations the Ion Chromatography staff is proposing to use as MDL for the SST Project the lowest Calibration level, ie., 0.250 ppm for halides and 0.500 ppm for the oxy-anions. Because accuracy falls off so badly below the calibration level, a conservative and realistic estimate of IDL would be at one half of the MDL. This offers a compromise between good accuracy and an adequate low level of detection.

14-243

000231

D05-008

Battelle
Pacific Northwest Laboratories

Project Number _____

Internal Distribution

Date September 8, 1993

To SG McKinley

From T Zyn

Subject Determination of Cr(VI) in SST Samples

Leach solutions from T-102, Core 55 received for analysis were analyzed according to Procedure PNL-ALO-227. Data is recorded in PNL LRB52921 pp.82-85. M&TE used were HP8451A spectrophotometer, WB76705 and Brinkmann 672 Meter, WB76896.

A blank was provided and analyzed with the sample set and one of the duplicate samples was post spiked. No prespiked sample was provided and was thus not analyzed.

ALO Number	Customer Ident.	Cr(VI)		Spike Rec.
93-08755-C-1	T-102 Core 55	741	RPD.	106%
93-08755-C-2	T-102 Core 55	745	(1%)	
93-08755-C-3	T-102 Core 55 Blank	<100		

Results are in µg/g. A nominal sample weight of 1.5g was used to calculate the blank value. Blank values are based on the method detection limit of 2µg Cr(VI), the nominal weight as indicated and the same sample aliquot size as used for sample analysis, although a larger aliquot was actually analyzed with no Cr(VI) detected. Using the actual volume used the blank value calculates at <13 µg/g. Sample results are based on sample weights and volumes as prepared in the 325-B Hot Cells.

The RPD value for the duplicata analyses is shown next to the results.

If there are questions regarding the results please contact MC Burt on 376-3762.

T Zyn 9-8-93

T ZYN, Sr. Technician
Analytical Chemistry Laboratory

Concur, DL Blanchard 9-9-93

DR-93-038 addressed the issue of IDL/MDL levels formatted.
9/10/93

DR-93-041 addressed the issue of no CCV, CCB and ICB analyzed in this run.
MC Burt
9/16/93

IA-244

000232

D07-003

Mercury by Cold Vapor Atomic Absorption Results

The Core 55 samples were digested and then analyzed by cold vapor atomic absorption (CVAA) for mercury following a modification of procedure PNL-ALO-213, "Mercury in Water, Solids, and Sludges by Manual Cold Vapor Technique." The modification has been documented by an instruction worksheet to the SAL operation and the Inorganic Analysis Group and merely changes the sample size, digestion volume, and heating method (i.e., from water bath to aluminum heating block). All core and QC sample digestions were performed in the SAL and the digestates transferred to the Inorganic Analysis Group for subsequent CVAA analysis. Since only limited quantities of samples can be digested with SAL at one time, the instrument calibration standards and calibration verification standards were digested by the Inorganic Analysis Group outside the SAL. This deviation is not expected to adversely affect the reported results since the independent QC samples digested in the SAL verify the calibration. Analytical results represent analysis of all the samples on two separate days; the result from the 8/25/93 run are reported in the summary table.

The RPD for the sample and duplicate of 46% indicate significant sample inhomogeniety (with respect to Hg) within the composite. At 5-8 µg/g, the mercury concentration is moderately high; however, nearly all the mercury has to be leachable for the material to be classified as toxic based on the mercury concentration. The Spike Blank "control" was recovered at 102%, indicating that the preparation and analysis operations were good. The spiked sample recovery was not recoverable since the concentration of the mercury in the samples was significantly higher than the spiking level.

The Hg analyses were performed twice, since the initial analysis used insufficient quantity of blank spike and process blank aliquots (i.e., 1 mL verses the standard sample aliquot of 10 mL). No CRA standard was analyzed; this is addressed in DR-93-033. The only sample QC to not meet acceptance criteria is the RPD; however, the pre-spike level did not allow evaluation of the spike recovery.

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